XXI B-MRS Meeting 2023

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Sociedade Brasileira de Pesquisa em Materiais

Proceedings of the
XXI B-MRS Meeting

Maceió, AL
2023

Revisão textual e gramatical: Resposanbilidade dos respectivos autores.

ISBN: 978-85-63273-54-3

Foreword

Ladies and gentlemen,

I am honored to stand before you today as the President of the XXI B-MRS Meeting and introduce this incredible gathering dedicated to the world of materials science and technology. This conference is not just an event; it is a celebration of knowledge, innovation, and the shared passion we have for advancing the field of materials science.

Our meeting reflects the profound impact that materials science has on our world. From the devices we use in our daily lives to the solutions that address global challenges, materials science is at the heart of progress.

Before we dive into the rich program that awaits us, I would like to take a moment to reflect on the importance of this gathering and the book that symbolizes our collective efforts.

This book, like our conference, is a repository of knowledge, a testament to human curiosity, and a tribute to the countless hours of research, collaboration, and dedication that each of you has invested in the field of materials science. Within these pages, you will find the latest discoveries, innovative technologies, and groundbreaking solutions that will shape the future of our discipline.

As we explore this book and the presentations throughout the conference, let us remember the essence of scientific discovery - curiosity. It is curiosity that has driven us to explore the properties of materials, to uncover the secrets of the universe at the nanoscale, and to engineer materials that have never existed before.

In our quest for innovation and sustainability, let us also embrace the idea of responsibility. Our discoveries and innovations have the power to change the world, and with that power comes the responsibility to ensure that our work benefits all of humanity and our planet. Sustainability is not an option; it is an imperative that must guide our research and our actions.

Throughout this meeting, you will have the opportunity to engage with leading experts, share your own research, and forge collaborations that will shape the future of materials science. The connections you make and the knowledge you gain here will not only advance your careers but will also contribute to the collective progress of our field.

I want to express my sincere gratitude to the organizing committee, the volunteers, our sponsors, and exhibitors for their unwavering support. Your dedication and hard work have made this event possible, and we are deeply thankful for your commitment to the advancement of materials science.

In conclusion, as we embark on this intellectual journey over the next few days, I encourage you to approach each presentation, each conversation, and each idea with an open mind and a spirit of collaboration. Let us use our knowledge and our passion for materials science to drive innovation and sustainability, and let us be stewards of the responsible use of our discoveries.

With that, I officially introduce the book of the XXI B-MRS Meeting, a testament to our collective pursuit of knowledge and progress.

Thank you, and let us embark on this exciting journey together. Enjoy the conference!

Carlos Jacinto da Silva Conference Chair

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Summary

PL-Plenary Lectures

Advanced Materials for Energy Efficient Printable Electronics

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With the growing smartness in electronic manufacturing worldwide, printed electronics capture attention because of their ability to overcome the limitation of traditional high-cost manufacturing approach which is mostly based on rigid silicon substrate. Moreover, has a great potential to offer biodegradable and recyclable solutions, by choosing low-cost substrate for printing devices, that may be recycled and/or naturally degraded in nature. This is a way forward to minimize the electronic waste (e-waste) caused by the ever-increasing number of disposable electronic devices. Relevantly, "printing electronics on paper" technology is rapidly developing in both research and electronic industry fields during the last decade. [1] For a plethora of applications, the printed devices and systems on foils require to be powered autonomously. To do so, within a sustainable approach green energy source should be exploited, such as photovoltaics thermo and mechanic energy harvester exploiting as much as possible the different materials performances as well design and architecture of the same. The development of stable, cost-effective, non-toxic, and eco-friendly printable devices for energy powering electronic printed devices on foils, such paper that are extremely lightweight, affordable, readily customizable, thin, flexible, and disposable are discussed in this presentation.

Carbon-based materials

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The carbon nanotube was the paradigmatic material of Nanoscience and Nanotechnology in the nineties. Despite the huge expectations created by its potential applications, few were confirmed. A similar expectation was experienced with studies on graphene, including the replacement of silicon in nanodevices, which is far from being a reality. However, the study of the properties of nanotubes and graphene has brought spectacular results from the pointof view of basic research, which explains the large number of groups dedicated to these subjects. Today, research on low-dimensional materials is diversified including, among others, TMDs, silicenes, phosphorenes, etc. this seminar, I will present a review of our research activity focused on carbon-based materials, starting with diamond-like carbon films, targeting applications such as protective coatings, passing through nanotubes, graphene, and quantum dots, with applications in sensors.

Computational Modeling and Design of OLED Emitters Based on Earth-Abundant Elements

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To achieve 100% internal quantum efficiency in electroluminescent devices, all singlet and triplet excitons must be harvested. The design of novel OLED emitters therefore requires detailed knowledge about excited-state decay pathways and transition probabilities including singlet-triplet interconversion processes. Quantum chemical methods can provide information about the properties of electronically excited states and their couplings and thus aid in understanding the underlying mechanisms. In our group, the DFT/MRCI method, a density functional theory based multireference configuration approach, and the associated property programs SPOCK, DELTA and VIBES are employed for this purpose. As the talk will show, these methods have proven to be well suited for evaluating spectra as well as intersystem crossing (ISC), internal conversion (IC) and radiative rate constants required for modeling the excited-state decay pathways of OLED emitters.

In search for bright and operationally stable electroluminescent emitters, the focus recently shifted from the well-established phosphorescent Ir^{III} and Pt^{II} emitters to Cu^{I} coordination compounds and metal-free donor-acceptor systems based on thermally activated delayed fluorescence (TADF). Cu is much more earth abundant than Ir or Pt and hence much less expensive. Moreover, due to the filled 3d shell, dissociative metal-centered (MC) d-d* excited states are energetically not low-lying in Cu^{I} complexes. Our computational studies suggest that linear and three-coordinated Cu^{I} carbene complexes with low-lying ligand-to-ligand charge transfer (LLCT) triplet states typically emit TADF whereas complexes with T_1 states of metal-to-ligand charge transfer (MLCT) character are phosphorescent.

Like Cu^{I}, Zn^{II} possesses a d^{10} configuration in the electronic ground state. Zn complexes might therefore qualify as TADF emitters as well. The low-lying states of newly designed Zn^{II} carbene dithiolates have predominantly LLCT character. In conjunction with non-negligible spin-orbit coupling (SOC) due to participation of sulfur orbitals in the electronic excitation, the small singlet-triplet energy gap ΔE_{S-T} leads to efficient triplet-to-singlet up-conversion.

TADF in metal-free organic through-bond and through-space charge-transfer (CT) systems often follows a different mechanism. These compounds have small ΔE_{S-T} values as well, but the mutual SOC of their S_1 and T_1 states is tiny. Spin-vibronic interactions with energetically close-by local excitations are found to essential for promoting the reverse ISC in metal-free compounds.

Graphene and Related Materials: From Production to Applications

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Disruptive technologies are usually characterised by universal, versatile applications, which change many aspects of our life simultaneously, penetrating every corner of our existence. In order to become disruptive, a new technology needs to offer not incremental, but dramatic, orders of magnitude improvements. Moreover, the more universal the technology, the better chances it has for broad base success. Significant progress has been made in taking graphene and related layered materials from a state of raw potential to a point where they can revolutionize multiple industries. I will overview the progress done thus far and the future roadmap.

Innovations in Materials and Technologies to Face New Threats in Water Treatment

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Providing fresh water in a sustainable and affordable manner is one of the greatest global challenges of our society, where over 2 billion people suffer from water scarcity. These estimations are projected to continuously increase owing to the growth of population, water pollution, climate change, and yet the impacts of COVID-19 pandemic. Despite the maturity of many technologies to assure water availability (providing fresh water and depollution used wastewater), these are mostly based on energy-intensive processes integrated in large and centralized infrastructures that are inaccessible to off-grid communities. Therefore, current challenges in water treatment are linked to the need for developing effective solutions to face emerging pollutants, capable of assuring a feasible economic upgrade of existing plants.

Conventional biological treatments are assisted with tertiary processes such as adsorption to improve the efficiency of the treatments. However, the cost associated to the regeneration of the spent adsorbents still represents a major limitation for massive implementation. On the other hand, advanced oxidation processes have become an interesting alternative for the simultaneous removal and degradation of pollutants. However, innovations on new materials based on sustainable approaches that can be integrated into physical, chemical, and biological processes to treat all types of waters are needed and require global research efforts.

This lecture will focus on the application of advanced adsorption, electrochemical and photocatalytic oxidation processes based on porous carbons, semiconductors and their hybrids combined with processes based on renewable energies for the removal and/or degradation of emerging pollutants in water. Examples will cover our research activities at lab-scale, as well as various case studies at pilot scale on the application of these technologies and materials to wastewater from industrial and treatment plant facilities origins.

These research activities have received funding from the European Union’s Horizon 2020 research and innovation program under the grant agreement № 776816 (Project Ô), the European Research Council (grant 648161, PHOROSOL) and Région Centre Val de Loire (grants MOSAIK, PRESERVE, MATHYFON).

Luminescent nanothermometers in biomedicine: past, present and future

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Temperature is crucial in cell and tissue dynamics. Because of that, temperature has emerged as a physiological parameter that can be potentially used for advanced diagnosis and treatment of different diseases ranging for cardiovascular misfunctions to alterations in the nervous system. Such advances require the development of new methods capable of remote thermal sensing in biosystems (from individual cells to organs and tissues). Unfortunately, conventional methods such as classical thermometers of thermographic cameras fail to provide such thermal readouts.

A luminescent nanothermometer (LNTh) is defined as a luminescent nanoparticle whose luminescence properties are strongly temperature dependent. In this case, remote thermal reading just requires an adequate analysis of the luminescence. LNThs have been applied in different fields ranging from microelectronics to nanofluids. But it is in the field of biomedicine where LNThs have resulted of most relevance. As I will summarize in this talk, LNTHs have been demonstrate capable of accurate thermal sensing in individual single cells, of in vivo early diagnosis of tumor development and of providing control to in vivo thermal treatments. I will include a critical analysis of the future of the field, the challenges that LNThs are facing and the actual possibility of transferring luminescent nanothermometry to the clinical world.

Porous nanomaterials for capturing, sensing andreleasing

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Design and realization of materials able to be biocompatible, degradable and programmed to target only desired organs and/or cells is a major challenge that involve several disciplines. We have for several years focused on organosilica nanoparticles able to break once they are internalized in cells and showed that it is possible to change morphology, and porosity. Very recently we have shown that cage type structure of small dimension (20 nm) can escape macrophage filtering [1] and reach the tumor site. Such findings have been tested to study the therapeutic effect of such nanocarriers for fighting the malignant pleural mesotelioma. In vivo data showed an interesting 50% reduction of tumor growth when mice were treated with a Pt(IV) prodrug, entrapped in the nanocages, at an equivalent dose of the free platinum complex [2]. However silica nanoparticles are also able to become responsive to biological input when synthetic nucleic acids and analogs as constitutive components of the organosilica structures [3]. The system can be programmed to be more dynamic and responsive by designing supramolecular organo-silica systems based on PNA- derivatives that can self-assemble through direct base paring or can be joined through a bridging functional nucleic acid, such as the ATP-binding aptamer [4]. Finally the capture of small metabolites such as the neurotransmitters serotonin and dopamine with unprecedented affinity and selectivity even in saline biofluids, is discussed using microporous hybrid materials. In particular zeolite-based artificial receptors which capture the analytes have been engineered with fluorescent reporters and results in biofluids are illustrated [5].

I wish to thank the European Union’s Horizon 2020 research and innovation programme (grant agreement No 964386 - FET Open Mimic-Key project).

References

1ACS Nano 2021, 15, 9701-9716
2Adv. Healthcare Mater. 2023,
3J. Am. Chem. Soc. in press.
4J. Am. Chem. Soc. in press.
5Adv.Mater. 2021, 33, 2104614

A-Artificial intelligence applied to materials science

Combining the Power of High-Throughput ab initio Calculations and Machine Learning towards Materials Informatics

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The progress in first-principles simulation codes and supercomputing capabilities have given birth to the so-called high-throughput (HT) ab initio approach, thus allowing for the identification of many new compounds for a variety of applications (e.g. lithium battery and photovoltaic). As a result, a number of databases have also become available online, providing access to various properties of materials, mainly ground-state though. Indeed, for more complex properties (e.g., linear or higher-order responses), the HT approach is still out of reach because of the required CPU time. To overcome this limitation, machine learning approaches have recently attracted much attention in the framework of materials design.

In this talk, I will review recent progress in the emerging field of materials informatics. I will briefly introduce the OPTIMADE API [1] that was developed for searching the leading materials databases (such as AFLOW, the Materials Cloud, the Materials Project, NOMAD, OQMD, ...) using the same queries. I will introduce the MODNet framework and its recent developments for predicting materials properties [2-3]. This approach, which is particularly well suited for limited datasets, relies on a feedforward neural network and the selection of physically meaningful features. Finally, I will illustrate the power of materials informatics, combining high-throughput ab initio calculations and machine learning, through a few recent examples.

References
[1] C.W. Andersen et al., Sci. Data 8, 217 (2021).
[2] P. P. De Breuck, G. Hautier, and G. M. Rignanese, npj Comput. Mater. 7, 83 (2021).
[3] P. P. De Breuck, M.L. Evans, and G. M. Rignanese, J. Phys.: Condens. Matter 33, 404002 (2021).

Engineering Soft-Matter with Agent-Driven High-Throughput Experiments

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Artificial intelligence (AI), when paired with laboratory automation, can greatly accelerate materials optimization and scientific discovery. For example, it can be used to efficiently map a phase-diagram with intelligent sampling along phase boundaries, or in 'retrosynthesis' problems where a material with a target structure is desired but a synthetic route is not known. These approaches are especially promising in soft matter and polymer physics, where design parameters (e.g. chemical composition, MW, topology, processing) are vast and where properties and function are intimately tied to molecular design features. However, for AI algorithms to operate efficiently in these spaces, they must also be 'encoded' with relevant domain expertise specific to the problems being tackled. This talk will cover recent advances in hardware and software tools for accelerated materials optimization for polymers and soft matter systems. Finally, it will outline remaining challenges in practical implementations and identify future opportunities for research.

Graph recommender system for ionic substitution: Discovering new materials

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Traditional chemical heuristics have been very important for discovering new inorganic compounds in the past, but as they are usually based on patterns observed on a limited or specific set of materials, they are not generalizable as once thought. Today, as one can access the data of hundreds of thousands of inorganic materials present in databases, it has become frequent that many Material Science projects have become data-driven, i.e., they use large amounts of materials data and statistical methods, such as Machine Learning(ML), to uncover new patterns and explore the phase space of compounds. In the ML context, recommender systems are widely used in various scientific and nonscientific fields. For instance, in the e-commerce area, these systems recommend users new products by analyzing their past purchased products or by collaborative filtering: a system where a heterogeneous network/graph composed of users and items and their interactions is used as input to recommend new products to users. By using this same principle framework and OQMD''s materials database, we were able to build an analogous graph with the elements in the periodic table together with the set of all described crystal structures'' sites as two classes of entities. The relationships between the two classes are given by the occupancy ofa site by an element and it is weighted by the compound''s thermodynamic stability. The heterogeneous graph is used to build an embedding space with a vector representation for each entity (elements and sites). These vector representations were then used as input to model a recommender system. In addition to finding new materials by uncovering new element-site occupancies, we were also able to analyze the embedding space and find interesting patterns in chemical similarities between elements and the local geometry of sites. We thank financial support from FAPESP and from the Materials Informatics INCT (CNPq).

Machine learning and Natural Language Processing in Materials Sciences

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The advent of large language models, as those used in tools such as ChatGPT, is bound to transform the way the scientific literature is mined and how knowledge discovery will be done in the future. While this applies to all scientific areas, specific tools for materials sciences are already available, which are capable of providing unprecedented results in terms of the identification of materials and their application. In this lecture, a discussion will be made of data analytics approaches, including natural language processing capabilities with deep learning, which can be implemented according to a pipeline of tools combining information visualization and complex networks. With the latter networks, one may probe the landscape of research areas, where papers from a given topic can be clustered into communities whose characteristics can be analyzed in detail. Also to be discussed is the possibility of leveraging multimodal data from distinct sources to produce surveys of the literature, and develop monitoring systems for diagnostics and surveillance. An example will be shown of the use of machine learning to process sensing data and clinical information for diagnosis.

A machine learning-enhancement for portable electrochemical detection of viruses.

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Biosensors are small analytical devices which converts a biological response into a quantifiable and processable signal. Among the different methods available in biosensing, electrochemical detection presents several advantages such as high sensitivity, easy-of-operation, cost-effectiveness and miniaturization capability. The current study proposed innovative machine learning-enhanced electrochemical detection of enveloped viruses such as rabies virus (RABV) and SARS-CoV-2. Portable detection was achieved by using an LMP91000 IC portable potentiostat circuit and a microfluidic sensor. Data were collected in collaboration with Campus Fiocruz the Atlantic Forest - RJ and Pasteur Institute - SP. Voltammetry results for rabies detection were used in data-mining process, feature extraction was performed using electrochemical parameters to build a dataset with uncovering patterns and other valuable information. Several statistical analysis and visualization technique were conducted to evaluate the constructed electrochemical dataset. Correlation-based algorithms, recursive feature elimination and feature importance were used for feature selection and evaluated by SHapley Additive exPlanations (SHAP) values. We experimented with several ML classifiers, namely support vector machine (SVM), Gaussian Naive Bayes (GNB), multi-layer perceptron (MLP), k-nearest neighbors (KNN), logistic regression (LR) and ensembled methods, and evaluate our findings in terms of different evaluation metrics such as recall and precision. Our analysis showed the best score of metrics for k-NN classifier for diagnostic tasks for rabies virus. The trained model will be implemented on ATmega328 microcontroller with embedded programming. Our study shows the potential of ML models in the field of electrochemical biosensor development with focus on parameter extraction and selection based on electroanalytical analysis, which can be extended to develop new tools for disease diagnosis in the future.

First principles physics-informed neural network for quantum wavefunctions and eigenvalue surfaces

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Physics-informed neural networks have been widely applied to learn general parametric solutions of differential equations. Here, we propose a neural network to discover parametric eigenvalue and eigenfunction surfaces of quantum systems. We apply our method to solve the hydrogen molecular ion. This is an ***ab initio*** deep learning method that solves the Schrödinger equation with the Coulomb potential yielding realistic wavefunctions that include a cusp at the ion positions. The neural solutions are continuous and differentiable functions of the interatomic distance and their derivatives are analytically calculated by applying automatic differentiation. Such a parametric and analytical form of the solutions is useful for further calculations such as the determination of force fields. Acknowledgement Fapesp process 17/18139-6

Machine learning as a tool on multiscale materials modelling: building bridges over scales

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Recently, computational materials modelling has been successfully used to understand the fundamental mechanisms underlying materials phenomena and processes. Strategies based on computational-guided experiments allow us to optimise and design materials (composition, morphology and processes). Important achievements in the discovery and design of materials through multiscale molecular simulations have been accomplished in our group, coupling first-principles calculations, molecular dynamics and lattice Boltzmann methods (1-4). However, a hierarchical multiscale approach represents a considerable challenge in modelling physical phenomena ranging from molecular to micro to macro scales. A key issue is that physical-chemistry phenomena in materials can occur on different scales. Here, we will share our strategies to incorporate machine learning methods to bridge scales over multiscale materials modelling. The case studies range from i) screening of catalyst and, ii) the development of interatomic potential energies based on First Principles Calculations and Molecular Dynamics, iii) fluid-fluid interfaces at the atomic scale, and iv) the prediction of pressure and velocity fields on mesoporous simulations usingLattice Boltzmann methods. The applications range from the CO2 mineralisation processesof CO2 and CO2 reduction towards value-added products and fluid flow through porous media. A bonus is using machine learning-based models coupled with virtual reality tools towards an interactive materials design on the fly.

(1) CR Miranda et al., Journal of The Electrochemical Society 169 (11), 116505, 2022
(2) Unraveling fluid-fluid interfaces through molecular simulations and machine learning, DN Lin, A Kirch, CR Miranda, 2022 Goldschmidt Conference
(3) CAF Salvador, BF Zornio, CR Miranda, ACS App. Mat. Interfaces 12, 56850 (2020)
(4) A Kirch, YM Celaschi, JM de Almeida, CR Miranda, ACS App. Mat. Interfaces 12, 15837 (2020)

CRM thanks RCGI, Fapesp and CNPq.

Machine Learning-Assisted Exploration of 111-Type 2D Perovskite Structures for Photovoltaic and Optoelectronic Applications: A High-Throughput Screening Approach

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In this study, we harnessed machine learning to scrutinize approximately 10 million 111-type 2D perovskite structures with the formula $A_3B_2X_9$for photovoltaic and optoelectronic applications. We examined six A-site cations, 23 B-site cations, and five anions, adhering to the stoichiometry $A_{6-2x}A^2{}_{2x}B_{4-y}B^2{}_yX_{18-3z}X^2{}_z$and confining compositions to halides.We applied the M3GNet graph neural network to ascertain optimal configurations for each composition, resulting in 1.8 million unique, lower-energy compositions. Subsequently, we trained a Materials of Optimal Descriptor Network (MODNet) model on 1.5 million materials from the Open Quantum Materials Database (OQMD) to predict stability based on compositions, eliminating those with a convex hull distance exceeding 0.2 eV/atom. This process yielded approximately 100,000 potential stable compositions.To further refine our selection, we utilized an ensemble of MODNet models trained on the entire OQMD database and exclusively on halides for formation energy and band gap calculations. This method enabled the identification of promising candidates with low band gaps and enhanced stability, ideal for photovoltaic and optoelectronic applications. Our approach showcases the remarkable capabilities of advanced machine learning techniques in delving into extensive chemical spaces that are impractical for expensive ab-initio calculations and exceedingly challenging for experimental synthesis. This methodology has the potential to significantly expedite the discovery and design of novel materials in the photovoltaic and optoelectronic domains contributing to advancements in perovskite materials research and fostering progress in renewable energy technologies. Furthermore, this approach can be tailored to address a wide range of research challenges in the broader field of materials science.

Acknowledgements:CNPq, CAPES, CESUP (in Brazil), F.R.S.-FNRS, CÉCI (in Belgium).

Machine learning-assisted MD simulation of melting in superheated AlCu crystal validates the Classical Nucleation Theory

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We performed comprehensive molecular dynamics (MD) simulations of spontaneous melting of a superheated AlCu crystal under atmospheric pressure at five temperatures, covering a range of T/T_L=1.1-1.3, where T_L is the liquidus temperature. Two realistic AlCu models were investigated: one described by the modified embedded atom method (MEAM) and the other by an interatomic potential generated by an artificial neural network machine learning (ML) approach, extensively trained on a massive *ab initio* dataset of liquid and crystal configurations. The temperature dependencies of the mean nucleation time, nucleation rate, critical nucleus size, work of formation, and nuclei growth rate were determined by MD simulations. Using these simulation-generated data, the theoretical nucleation rates were calculated by the Classical Nucleation Theory (CNT) with no fitting parameters. We found excellent agreement between the theoretically and MD-computed liquid nucleation rates for both MEAM and ML crystals. Notably, the computational value of the effective solid-liquid interfacial free energy aligns with its recent experimental measure. Moreover, the CNT qualitatively and quantitatively described the underlying details of liquid nucleation in our ML solid, accurately reproducing the kinetic factor and the size, formation energy, and growth rate of the critical nuclei. Thus, the melting of the AlCu model created through ML-processed quantum calculations, that is, not relying on hand-crafted interatomic potential functions, was successfully described by the CNT phenomenological formalism, without any adjustable parameters.

Acknowledgments: A. Tipeev acknowledges the São Paulo Research Foundation, FAPESP, Brazil, grant # 2022/05837-5. E. Zanotto also thanks FAPESP (contract CEPID # 2013/07793-6) for the financial support to this research. R. Ryltsev was supported by the Ministry of Science and Higher Education of the Russian Federation (state assignment for IMET UB RAS # 122013100200-2).

Natural language processing towards a data-oriented design approach for producing biochar and hydrochar

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To effectively apply biochar and hydrochar in various technologies that can enhance human life, it is crucial to comprehend their properties and characteristics, as they significantly impact their application. Understanding the correlations between experimental conditions and the resulting properties of these carbon-based materials can provide a better control of the synthesis process and final product properties. This could ultimately lead to a controllable design of biochar and hydrochar for use as CO2 adsorbents, soil amendments, and in other applications. Here we describe how natural language processing (NLP) can be used to extract relevant data from thousands of peer-reviewed scientific papers describing the synthesis conditions and properties of biochar and hydrochar. With the collected data, machine learning (ML) algorithms could be employed to identify previously unknown correlations between synthesis and properties, such as: (i) how the carbonization temperature can influence the material final surface area (SA); (ii) which are the most suitable biomass precursor to produce biochar and hydrochar with the largest adsorbent capacity; and (iii) what processing steps must be performed to achieved a high C/H ratio. By adopting data-driven and statistically supported approaches, we show that there is a great potential to change traditional manufacturing paradigms and accelerate informed decision-making on the design of carbon-based materials, especially those produced from renewable sources.

Prediction of self-healing properties of concretemodified with fibers and bacteria

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Self-healing concrete has been studied for decades as an alternative material to overcome problems such as cracks and low durability of conventional concrete. However, laboratory experiments can be costly and time-consuming. Thus, predictive computational models that can estimate the properties of cementitious materials are being increasingly applied to investigate and develop new types of concrete. In this work, Machine Learning (ML) models were developed to predict and analyze the repairing rate of the cracked area and capillary water absorption in concrete modified with alkali-resistant bacteria, polyvinyl alcohol (PVA) and polypropylene fibers (PP). The empirical datasets used for training the models were produced and published by Feng et al [1] and Su et al [2]. The implemented ML algorithms were Simple Linear Regression (LR), Multiple Linear Regression (MLR), Support Vector Machine (SVM) and Random Forest Regressor (RFG). The results show that among the algorithms, the most efficient and accurate were the SVM and the RFG. In addition, the model trained with the dataset containing the variables related to the rate of the cracked area obtained better prediction performance. RFG was more efficient and accurate in predicting the cracked area rate (R2 = 0.98, RSME = 0.0009), followed by SVM (R2 = 0.95, RSME = 0.0028), while for predicting water absorption, SVM showed better performance (R2 = 0.76, RSME = 0.0566), followed by the RFG (R2 = 0.71, RSME = 0.0651). Thus, the results show that ML can be a valuable tool for the AI and Data-Driven development of new and improved concrete.

Acknowledgments:
This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001

References:
[1] FENG, Jianhang; et al. Construction and Building Materials, vol. 228, p. 116810, 2019.
[2] SU, Yili et al. Cement and Concrete Composites, v. 116, p. 103896, 2021.

Supervised machine learning models developed topredict the properties of biocompatible Ti alloys

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Titanium (Ti) and its alloys are considered suitable metallic materials for biomedical applications, mainly for bone implants. Beyond good combination of properties that attend different biocompatibility requirements, Ti alloys can achieve values of elastic modulus which approach the values presented by human bone [1]. However, due to the variability in the properties achieved with the addition of different chemical elements, Ti alloys continue to be studied for searching the chemical composition which results in the lowest elastic modulus. In this way, with the availability of reliable database in the scientific literature for different chemical compositions of Ti alloys already experimentally characterized, as well as, with the recent published architectures of promising artificial neural network (ANN) models [2], three new models of ANN were created in the present work aiming to aid the development of new Ti alloys for biomedical application. Thus, the weight percentages of Mo, Nb, Ta, Zr and Sn were considered as input neurons, while the elastic modulus was considered as output neuron. Different activation functions were applied to the neurons in the hidden layers, in addition to different standardizations performed on the data before modeling. The results showed that the model with the Sigmoid function and with the standardization between zero and one achieved good convergence, registering the root mean squared error equal to 10.3 GPa, even with few epochs in the training. Therefore, ANN modeling showed promise for greater use in the design of new Ti alloys.

References

[1] S. S. Sidhu, et.al, Mater. Sci. Eng. C, 111661 (2020).
[2] C. T. Wu, et.al, Materials Today, 34, 41-50 (2020).

Synthesizing double halide perovskites with machine learning prediction

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Lead perovskite solar cells have been successful in achieving an efficiency of over 25% in just over a decade of research. Once the lead perovskites instability remains a challenge, there has been a growing interest in more stable lead-free perovskites, such as double halide perovskites. [1] However, the synthesis process is complex due to the numerous compositions and conditions involved. To improve this search, a multi-target machine learning study was conducted to analyze previous experimental data and predict synthesis parameters. The predictions were used to synthesize two new double halide perovskites (Cs2CuLaCl6 and Cs2AgLaCl6) by the hot injection method. We created a database to describe the synthesis and properties of the perovskites, which had 10 features and 164 observations after cleaning and filtering. Three algorithms that support the multi-target approach were applied: decision trees, random forest, and neural networks. The five outputs used to train the algorithms and predict conditions included reagent quantities, time of reaction, and bandgap. We chose random forest as the final model and synthesized the perovskites, by the hot injection method, based on its predictions. By the precipitate XRD diffraction patterns, we observed the formation of cesium chloride. On the UV-Vis spectra of the Cs2CuLaCl6 and Cs2AgLaCl6 supernatants, the indirect bandgaps were 3.5 and 3.4 eV, respectively. These values are close to the predicted bandgaps of the perovskites by the machine learning model: 3.8 and 3.7 eV, respectively. The direct bandgaps (2.7 and 2.9 eV) may indicate possible intermediate formations. We must carry out further characterizations to confirm the formation of the perovskites. Future research will focus on optimizing the synthesis process and testing the materials in solar cells to evaluate their device performance.

Acknowledgements:
We thank PRH-ANP and CNPq for the financial support.

References:
[1] Energy Environ. Sci., 13, 4691-4716, 2020.

A combined DFT and machine learning study to understand catalyst and solvent effects on the conversion of CO2 into ethanol

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The conversion of CO2 into valuable fuels and chemicals has drawn significant attention due to the possibility of tuning the fuel yield and selectivity by changing the catalyst. Regarding this, decades of advances in theoretical and experimental methods have produced a compendium of complex data containing valuable information on materials that can and should be used to optimize reactions. In this work, our approach combines machine learning methods and DFT data to screen catalysts and find the best candidate to use as a catalyst to CO2 conversion. As previously reported, the C-C bond formation is the determinant step to obtain C2+ products of higher commercial interest, such as ethanol. The C-C bond coupling is usually related to intermediates, such as C* and CO*. The elemental reactions from gaseous CO2 to these intermediates and side reactions can be used to find the most favorable reaction pathway. The side reactions can lead to usually undesired products such as methane and methanol. As theoretical information from each elemental reaction can be found in the literature for many catalysts, it can be used to feed a dataset and train a machine-learning algorithm. We are also performing DFT calculations to improve the dataset, considering catalysts that already showed good experimental results, mixtures of metals and single-atom supported by usual substrates. The selected catalysts will be both theoretically and experimentally verified. As the solvent and the alkaline metals cations were previously demonstrated to play an essential role in these reaction pathways, they will also be considered.

Artificial neural networks applied in the catalytic process of urease and conjugated polymer thin films

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The increasing demand for molecule detection and quantification leads to the study and development of several techniques in biosensor production. The production of these devices is essential to have a model that can unravel and facilitate interpreting the obtained data. However, catalytic processes generally involve several variables, creating a non-linear system that conventional methods may not model [1]. Therefore, Artificial Neural Networks (ANNs) have claimed attention due to their capability to model data from several sources, even non-linear ones, by mimicking the human brain's learning process. In summary, the present work tries to integrate experimental data and simulations using ANNs. The catalytic films were made by the adsorption of urease in conjugated polymers using the spin coating technique, and the catalytic activity was obtained by an in-situ reaction, as described in the literature [2]. In the model development, the multilayer perceptron ANN was used since 12 architectures were created varying the variable disposition, and 45 structures were created for each architecture varying the number of neurons, algorithms of optimization, and transfer function. As a result, the best structure performed the testing step with a Pearson's correlation coefficient (ρ) of 0.9980 and objective function in the order of 10-5. Simulations were performed with new data to evaluate the efficiency of the trained ANN. The results achieved a mean absolute percentage error below 4% with good reproducibility of the experimental data.

Acknowledgments:
This work was supported by INEO, CNPq (310647/2020-7), and FAPESP (grant number 2020/04427-2, 2014/50869-6).

References:
[1] S. Tajik, S. Ayoubi, F. Nourbakhsh. Environmental Engineering Science. 29(8), 798-806. (2012).
[2] C. G. de Jesus, da R. da R. Rodrigues, L. Caseli, L. O. Péres. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 654, 130-136. (2022).

Boosting MODNet Capabilities: Fast Featurization, Gradient Boosting Augmentation, and Pretrained Model Synergy for Materials Science Predictions

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Machine learning models, particularly feature-based and graph neural network models, have propelled materials science by expediting new material discovery and property optimization. MODNet (Material Optimal Descriptor Network) is a versatile feature-based framework adept at predicting a wide range of crystal properties. It utilizes a feedforward neural network and physically meaningful features, selected by an efficient relevance-redundancy algorithm. Benchmarking against the MatBench v0.1 test suite showcases MODNet''s effectiveness, particularly with smaller datasets. MODNetFast, a featurizer developed in this work, addresses the slow featurization issue in MODNet, which relies on MatMiner featurizers. It adopts an efficient featurization process using autoencoders and MEGNet models to compress and reproduce essential features. Additional features, such as OrbitalFieldMatrix and pretrained MEGNet model dense layers, are integrated to improve prediction accuracy and generalization. Consequently, this new featurizer reconciles the benefits of feature-based models, boosted by graph-based models'' accuracy, while accommodating larger datasets. Simultaneously, a gradient boosting augmentation technique has been implemented in MODNet. XGBoost models are trained on optimal features across the entire training data and subsets based on similarity to generate additional predictors. Although this method is prone to overfitting, it enhances predictions in several cases and is employed only when it reduces validation loss compared to the original model. In conclusion, MODNetFast effectively extends MODNet''s applicability to larger datasets and leverages information from pretrained models and complex features. Furthermore, the gradient boosting augmentation approach refines MODNet''s performance in specific cases, making the combined framework a valuable expansion of MODNet''s capabilities.

Acknowledgments: CNPq, CAPES, CESUP (in Brazil), FRS-FNRS, CÉCI (in Belgium).

Machine learning models for materials screening applied to CO2 reduction and obtaining value-addedproducts

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The chemical reduction of carbon dioxide (CO2), a primary contributor to global climate change, is a necessary process that has been studied extensively. There are proposals to transform CO2 into chemical products through catalytic routes. The goal is to convert this gas into value-added products, e. g., oxalate salts, oxalic acid, hydrocarbons and methanol, using metallic catalyst and metallic heterostructures. In these processes, many intermediate products can arise. The interaction between the adsorbates and surface influences the selectivity, and it would be possible to control the final products by controlling the adsorbability of chemical species. Thus, understanding the behavior of catalysts is fundamental to optimizing and controlling the reduction process. In this work, we used machine learning methods to predict the adsorption energies of different chemical species related to CO2 reduction on different catalysts. We implemented machine learning models using the database''s information of public repositories and useful libraries to represent the interaction between the adsorbate and the catalyst. Our results showed the robustness of machine learning models and the ability of these methods to speed up the screening materials to specific goals at a low computational cost [1]. We emphasize the capability of our model to predict the adsorption energy of ions on metallic surfaces. It has a special significance due to the difficulty of calculating the correct energy for charged systems by traditional atomistic simulations.

Acknowledgements:

The authors acknowledge RCGI, FAPESP (2020/15230-5), Shell Brazil and CNPq.

References:

[1] Amaral, Paulo HR, et al. Journal of The Electrochemical Society 169.11:116505 (2022)

Machine Learning prediction of Self-Healing Capacityof Concrete with the use of Synthetic Data

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Concrete is the most widely used material in civil engineering. It is inexpensive, durable and resistant. Although its advantages, concrete has also some disadvantages, such as cracking, which cause high permeability in concrete structures. Thus, life span of concrete is reduced as cracking increases. Therefore, there is a concern about fixing cracks before they comprise the concrete or even the entire building. Although crack fixing is often made by adding new cementitious materials over the cracks, self-healing concrete has been subjected to research for years, including self-healing in concrete aided by bacteria. Machine learning (ML) can assist the prediction and development of new concrete formulae enriched with bacteria. Moreover, ML can reduce the time and cost in the development of new self-healing concrete. In this work, ML models to predict the capacity of bacteria-rich self-healing concrete were developed. Data of bacterial self-healing concrete were collected from the literature. A total of 38 occurrences involving six bacteria were collected from the literature. All data were record into a CSV table. As machine learning algorithms often requires large amount of data [2], the data compiled from the literature were subjected as a source to synthetic data generation via the Synthetic Data Vault. After data generation, two ML models algorithms: Support Vector Machines (SVM) and K-nearest neighbor (KNN) were trained using repeated cross-validation to predict if each concrete formula would have self-healing capacity. SVM models were able to predict self-healing with an accuracy of 0.913 and KNN with an accuracy of 0.875. Therefore, we conclude that SVM and KNN can be further improved to predict self-healing capacity of concrete with high accuracy.

Using Neighborhood Cultivation Genetic Algorithm to Identify the Parameters of a Time Dependent Mechanical Model for PTFE Powder Pressing.

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Material Science is the study of the structure, properties and processing of materials. The use of Artificial Intelligence (AI), represented by the Neighborhood Cultivation Genetic Algorithm (NCGA) can reduce the number of tests demanded to identify complex mechanical behavior parameters. As an example, the uniaxial pressing of Polytetrafluoroethylene (PTFE) pellets may show time-dependent properties due to the rearrange of them, resulting in creeping and stress relief. It demands a numerical model capable of time-dependent representation of mechanical properties. PTFE is commonly used in applications when high working temperatures (up to 260 °C) and chemical stability are needed. The main disadvantages of PTFE in the industry are the costs associated with the manufacturing process and raw material production. In the melting state, PTFE presents a very high viscosity that requires a processing route based on pressing of the pellets followed by sintering. The identification of models and calibration of the parameters are very important for the numerical simulation of the process, and demands many experimental tests. However, this number may be reduced by using an AI algorithm as NCGA. In this work, uniaxial pressing tests were carried out in a close die. A max displacement was set for the piston, which velocity varied in each test (40, 4, 0.4 e 0.04 mm min-1). After pressing, a dwell time was used to observe stress relief effects. The experiments were modeled in AbaqusTM using Drucker-Prager/Cap (DPC) and the viscoplasticity model of Singh-Mitchell. To identify the viscoplastic parameters, the computational results were compared to the experimental ones and the Singh-Mitchell parameters were varied by the NCGA until achieve a good correspondence between results. This methodology reduced the number of experiments, in real and virtual world, and helped to the set-up of experimental variables. Acknowledgment FAPESP grant # 2021/14452-7.

B-Design of intelligent hybrid and composite nanosystems: from Concepts to Programmable Matter

Bacterial Nanocellulose-Based Electrochemical Biosensors: An Overview and Producing Graphene on the Spot

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Bacterial nanocellulose (BNC) has gained a promising role as an alternative to anti-fouling surfaces. BNC may form part of a support for immobilizing specific materials to widen its applicability as a biosensing device. The possibilities of modification methods and ***in situ***and ***ex situ***functionalization enable new BNC properties. With the new insights into nanoscale studies, we expect that many biosensors currently based on plastic, glass, or paper platforms will rely on renewable platforms, especially BNC ones. Recently, paper has become a simple and inexpensive platform for electrochemical detection creating a new generation of electrochemical paper-based analytical devices (ePAD), however. deposition of electrodes over paper can be difficult, but graphene production on site is simple if combining the production process with a CO_2laser scribing. This process results in a graphene porous material with a large specific surface area.

Design of catalyst modified electrode using organic-inorganic hybrid metal hydroxide salt monolayer nanoparticles

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Transition metal-based layered hydroxides are known as potential candidate catalysts for efficient oxygen evolution reaction. Since the finding about excellent electrochemical catalytic performance of exfoliated hydroxide nanosheets, many studies focus on the synthesis and catalytic use of hydroxide nanosheets/nanoparticles. In general, surface modifiers are necessary to synthe hydroxide nanosheets/nanoparticles to prevent aggregation and agglomeration. Surface modifiers will affect the electrochemical performance of hydroxides because electrochemical catalytic reaction will occur at the surface of the materials. However, there are scarce report. Here, we focus on the design of electrocatalyst modified electrode and effect of surface modifiers on the electrocatalytic performance of metal hydroxide nanosheets/nanoparticles.

Metal hydroxide nanosheets/nanoparticles were prepared based on our previous report.[1] Ethanolic solutions dissolving metal chloride and carboxylic acids were used as starting precursors. Addition of propylene oxide triggered pH increase and nucleation and crystal growth of metal hydroxide salt nanoparticles. Nanocolloids were collected after evaporation of residual propylene oxide. The Au electrode modified with self-assembled monolayer was dipped in nanocolloids to immobilize nanoparticles through interaction of organic groups. Electrocatalytic property of metal hydroxide salt monolayer nanoparticle was successfully measured and found to improve after electrochemical redox cycle. Compared with common metal hydroxide nanosheets, our metal hydroxide salt monolayer nanoparticles showed > 1000 times higher weight normalized catalytic performance. In addition, mesoporous Au electrode could be employed only for monolayer nanoparticles case, which enabled enhanced areal catalytic activity owing to the high specific surface area derived from mesoporous structure.

References

[1] N. Tarutani et al., ACS Mater. Lett., 2022, 4, 1430–1435

Hybrid nanomaterials for biomedical applications

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Advancements in the use of nanoparticles for biomedical applications have clearly shown their potential for the preparation of improved imaging and drug-delivery systems. However, only a few successfully translate into clinical practice, because the nanoparticle are uptaken by macrophages. We have recently reported disulfide-bridged organosilica nanoparticles with cage-like morphology, and assessed in detail their bioaccumulation in vivo. [1] The fate of intravenously injected 20 nm nanocages was investigated in both healthy and tumor bearing mice. Interestingly, the nanoparticles exclusively co-localize with hepatic sinusoidal endothelial cells, while avoiding Kupffer-cell uptake in both physiological and pathological condition. Such findings have been used to study the therapeutic effect of such nanocarriers for fighting cancer and in particular malignant pleural mesotelioma. We have stabilized, transported and intracellular released a platinumIV prodrug using the breakable nanocarrier. Its reduction, and therefore activation as anticancer drug, is promoted by the presence of glutathione in neoplastic cells that also causes the destruction of the carrier. In vivo data showed an interesting 50% reduction of tumor growth was observed when mice were treated with the PtIV, entrapped in the nanocages, at an equivalent dose of platinum complex. [2]In addition they are able to stabilize out of equilibrium species and transport them inside cells were they can be released and evolve towards the equilibrium state. [3] Both materials can be covered with a lipidic layer to improve the hemocompatibility and improve cell uptake rendering the nanomaterials biomimetic.

We wish to thank the European Union’s Horizon 2020 research and innovation programme under grant agreement No 964386 (FET Open Mimic-Key project).

[1] ACS Nano 2021, 15, 9701-9716
[2] Advanced healthcare materials, 2023, DOI: 10.1002/adhm.202202932
[3] J. Am. Chem. Soc. 2021, 143, 7681-7687.

Luminescent probes based on hierarchically structured lanthanide-doped NaREF4 nanoparticles

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The engineering of hierarchically core@multishell nanoparticles containing heterogeneous crystalline phases in different layers constitutes an important strategy for obtaining luminescent probes. The possibility of obtaining an opto-magnetic core@multishell nanoparticle capable of emitting from VIS to NIR by upconversion, dowconversion and downshifting processes is highly desirable, especially when its optical responses are dependent on temperature or magnetic field variations. Our research group has great knowledge on synthesizing hierarchically structured NaREF4 core@multishell nanoparticles of heterogeneous crystalline phases, cubic or hexagonal, applied to magnetic or photophysical properties, respectively. The crystalline and compositional hierarchies are satisfactorily obtained and confirmed by DRX, EDS mapping and HAADAF images, allowing the optimization of optical or magnetic properties in different nanoparticle regions. Optically active shells emit in the VIS or NIR regions after excitation at 980, 808, and 1550 nm, covering different biological windows. The nanoparticle emissions are affected by temperature, where ErIII upcoversion, for example, may be taken as a temperature probe in a temperature range of 77 K to 427 K. Dowshifting emission in the NIR region covers higher temperatures ranging from 427 to 633K. The core@multishell nanoparticle can also have its luminescence modulated by the application of an external magnetic field that at low temperature improves the emission intensities and promotes the unfolding of Zeeman''s doublets of the Kramer ErIII ion. We also probed upconversion as a function of time and power of Surface Acoustic Wave (SAW), especially for high power regimes and low temperatures, the optical response indicates a considerable local temperature increase. Therefore, the magneto-optical properties of nanoparticles reveal their versatile nature and applicability as magnetic field or temperature luminescent probes.

Molecular-Electronic Switches that Mimic Synapses

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Molecular electronic devices are highly complementary to traditional electronic devices and may lead, in principle, to novel functionalities that are very complicated to realize otherwise[1,2]. I will discuss our recent efforts to develop multi-functional molecular devices[3,4,5]. We developed a new type of an electrically driven molecular switch that can toggle between two different functionalities, that of a diode and memory.[5] This dual-functional switch resembles one diode-one resistor (1D-1R) resistive random access memory (RRAM) but defined within a single molecular layer greatly reducing the operatingvoltage (to just

Molecule/Framework orientation on inorganics for anisotropic electrical, optical and thermal properties

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Precise control of the material interface, i.e., lattice matching at the interface, is important for the fabrication of oriented thin films with sophisticated functionalities. In the field of inorganic materials, epitaxial growth by lattice matching at the interface has been deeply investigated and contributed to the development of semiconductor devices. We have been reporting unique organic-inorganic epitaxial interface on metal hydroxides, which affords orientation of organic molecules and the fabrication of oriented metal-organic framework (MOF) films[1-6]. Crystalline metal hydroxides have hydroxyl groups (M-OH units) regularly arranged on their surface of which alignment reflects their crystalline structure. The ordered array of hydroxyl groups defines the alignment of the organic molecules on the surface (organic-inorganic epitaxial interface), affording the fabrication of oriented MOF films [1]. Because the MOF micropores are aligned to the specific directions in the macroscopic scales in the oriented MOF films, thin films with anisotropic properties for advanced technologies including electronic, optical, and thermal devices can be achieved via a host-guest approach by an accommodation of functional guests (molecules, ions, or nano objects) into the pores. For example, oriented MOF films impregnated with fluorescent molecules exhibited polarization-dependent optical responses[3]. In this presentation, our resent progress on the fabrication and applications (unique optical, electrical, and thermal properties) of oriented MOF films will be reviewed.

References

1) Falcaro P., Okada K et al., (2017) Nature Mater., 16, 342. 2) Ikigaki K., Okada K et al., (2019) Angew. Chem. Int. Ed., 58, 6886. 3) Okada K et. al., (2020) Chem. Sci., 11, 8005. 4) Ikigaki K., Okada K. et al., (2021) ACS Appl. Nano Mater., 4, 3467. 5) Okada K. et al., (2021) J. Mater. Chem. A, 9, 19613. 6)Baumgartner B. et. al., (2021) Chem. Sci., 12, 9298.

Organic emitting compounds as smart molecular materials

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Light or electric pulses are commonly used as external stimuli in the field of smart materials to control the macroscopic behavior. At molecular level, the organization of molecules at the macro-scale as thin films or solids influences the color of the emitted light. Hence, a planned chemical design plays a pivotal role to tune the properties of the small compounds and then to the bulk. Among the chemical species, organic molecules offer a large palette of easily structural modifications due to their chemical versatility, which allows a fine tuning of their properties, thus of their applications. In particular, organic emitting compounds can be considered as smart molecular materials as effect of the changes on the photophysical and electrochemical properties upon specific interactions with other species or with themself. In this contributions, examples of molecular systems exhibiting modifications on their emission upon specific interactions, such as host-guest interaction,[1] aggregation-induced emission[2] and self-assembled monolayer[3] will be presented.

Acknowledgements:
Deutsche Forschungsgemeinschaft (DFG) for financial support

References:

[1] M. Hayduk, T. Schaller, F. C. Niemeyer, K. Rudolph, G. H. Clever, F. Rizzo, J. Voskuhl, Chem. Eur. J. 28, e202201081 (2022)
[2] J. Dubbert, M. Valtolina, A. Huber, T. D. Scherz, C. Wölper, C. G. Daniliuc, O. Filiba, S. Sen, I. Schapiro, F. Rizzo, J. Voskuhl, ChemPhotoChem 7, e202200169 (2023)
[3] S. Kudruk, E. Villani, F. Polo, S. Lamping, M. Körsgen, H. F. Arlinghaus, F. Paolucci, B. J. Ravoo, G. Valenti, F. Rizzo, Chem. Commun. 54, 4999 (2018)

Organic-inorganic composites and aerogels

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Bacterial cellulose (BC) is a biocompatible and nontoxic biopolymer that has been successfully used as a sustainable substrate for flexible organic light emitting diodes (OLEDs). Adding recycled polystyrene to transparent BC membranes new conformable OLEDs substrates are fabricated. Polystyrene found in expanded foam package waste isdissolved in d-limonene (a green solvent extracted from orange peels), sprayed on BC membranes and tested as substrates for OLEDs [1].

In another application the polystyrene/d-limonene solution was used to well disperse upconversion nanoparticles. The new ink was used in order to fabricate new QR-codes displayig the optical properties of the nanoparticles. Temperature sensing was demonstrated. [2]

Finally, we will show new aerogels organic-inorganic hybrids membranes composed of cellulose and MoS2. These membranes show promising behavior for application in in-flow water purification, representing a significant advancement in the use of selfsupported aerogel membranes for photocatalytic applications in liquid media [3]

Acknowledgements: Brazilian agencies, CNPq, CAPES, FAPESP and the National Institute of Photonics are acknowledged for the support.

References:

[1]-A.V .S. Cebrian et al, Development of Conformable Substrates for OLEDs Using Highly Transparent Bacterial Cellulose Modified with Recycled Polystyrene, Adv. Sustainable Syst. 2000258, 2021

[2]-F.E. Maturi et al, Sustainable Smart Tags with Two-Step Verification for Anticounterfeiting Triggered by the Photothermal Response of Upconverting Nanoparticles Adv. Photonics Res. 2100227, 2021

[3]-E.P. Ferreira-Neto et al, Bacterial Nanocellulose/MoS2 Hybrid Aerogels as Bifunctional Adsorbent/Photocatalyst Membranes for in-Flow Water Decontamination ACS Appl. Mater. Interfaces 12, 41627, 2020

Photoresponsive soft materials made by molecular self-assembly

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Self-assembly is emerging as a superior method to prepare responsive and adaptive nanomaterials. The structure and function of these materials is entirely determined by the dynamic and weak interactions of the constituent molecular "building blocks" of the material. Since the inherent interactions are weak, these versatile materials readily respond to even small changes and stimuli in their environment. Moreover, these materials are biomimetic and can contain large amounts of water, so that applications in biomedical technology are foreseen.

This lecture will highlight our recent work on self-assembled supramolecular nanomaterials that respond to light. In all these nanomaterials, molecular photoswitches are key components. The improved molecular design of photoswitches enables the bottom-up self-assembly of tailor-made functional materials and interfaces. Amongst others, light responsive hydrogels, foams, monolayers, adhesives and solids will be discussed.

References:
[1] L. Stricker et al. J. Am. Chem. Soc. 2016, 138, 4547-4554.
[2] C. W. Chu et al. Chem. Eur. J. 2019; 25: 6131-6140.
[3] L. Kortekaas et al. ACS Appl. Mater. Interfaces 2020, 12, 32054-32060.

Plasmonic hierarchical superstructures as highly efficient SERS substrates for applications in health and environment

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Currently there is a high demand for ultrasensitive sensors for both health and environmental applications, and both areas are closely related. The detection of contaminants in food, water, and in general in the environment is essential since many of these are usually harmful to health. On the other hand, the detection of cancer biomarkersin early stages of the disease is essential to have greater chances of success in its eradication. In recent years, plasmonic hierarchical superstructures have played an important role in the development of new sensors with high detection capacity. These materials can be used as SERS substrates, which base their detection principle on surface enhanced Raman scattering. In this talk, several strategies for the synthesis of plasmonic nano and superstructures will be presented, as well as the evaluation of their performance as SERS substrates for the sensing of model molecules, contaminants, drugs and cancer cell lines. Several synthesis strategies were used: chemical synthesis, electrodeposition, electrophoresis and galvanic replacement. Various nano and superstructures such as nanostars, nanoparticle-decorated nanodendrites, nanostructured hollow polyhedrons, among others, based on Au, Ag, Cu and their combinations, were synthesized. Some of the pollutants and biomolecules detected were: model molecules (rhodamine 6G, 4-aminothiophenol, crystal violet), emerging pollutants such as bisphenol A and triclosan, antibiotics such as tetracycline and vancomycin, and opioid analgesics such as tramadol. In addition, through adequate functionalization, it was possible to specifically sense a prostate cancer cell line (LNCaP). The results obtained show us the high potential of hierarchical plasmonic nano and superstructures for the development of ultrasensitive SERS substrates.

Simulation and Theoretical Studies on Nonequilibrium Processes of Soft Matter Near Interfaces

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In this talk, I would briefly summarize our main findings on the simulation and theoretical studies of nonequilibrium processsces of soft matter near distinct interfaces in the past 10 years. We focus on the physical aging of ultrathin polymer films at solid-vapor interfaces [1], the interfacial crystallization of polymers near solid-liquid-vapor interfaces [2], and the evaporation of binary solvents at liquid-vapor interfaces [3]. Given the computer simulations and dynamical mean-field theories, we provide physical explanations on some interesting phenomena, such as the slowing down of accelerated aging [1], the anomalous Ostwald ripening during the skin layer within the evaporative skin layer [2], and the evaporation-induced liquid expansion in binary solvents [3]. These findings might foster our fundamental understanding of the nonequilibrium processes of soft matter near interfaces and promote their practical applications.

Acknowledgements:
The author acknowledges the finacial support from the NSFC and DFG, and the collaborations from Professors of W. Hu, S. Napolitano, M. Müller, and C. Y. Li.

References:
[1] Q. Tang, W. Hu and S. Napolitano, Phys. Rev. Lett., 112, 148306 (2014)
[2] Q. Tang, M. Müller, C. Y. Li and W. Hu, Phys. Rev. Lett. 123, 207801 (2019)
[3] Q. Tang and M. Müller, Phys. Rev. Lett. 126, 028003 (2021)

DESIGN AND SYNTHESIS OF PROTEIN-CARRYING NANO-VEHICLES FOR LIGHT-INDUCED RELEASE

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Protein transport in a nano-vehicle (NV) is envisaged for local treatments as the protein is protected from degradation during transport and once inside the target cell can be released by some external stimulus. Designing, modelling, and synthesizing complex nano-objects that integrate signals and functions is an open frontier and is a very active research topicwhere formidable advances have been made. However, the synthesis of these NVs still presents numerous challenges given the complexity of the system and the difficulty ofcontrolling the kinetic parameters.

Here we present the design and synthesis of mesoporous silica (MPS)-based NVs with a gold core (Au/mSiO2) that meet the basic requirements of adequate to pass through a cell wall, high loading capacity, stability during transport, and stimulus-triggered release of the protein at the target. In this presentation, we will focus on the synthesis pathways that provide reproducible and monodisperse Au/mSiO2 particles with pores suitable for trappinga protein by non-covalent interactions, diameter less than 100 nm, and thickness of the silica layer such that the protein can be released by plasmonic heating.

One-pot and seed-growth pathways were deeply investigated. Even one-pot is less time-consuming and scalable, Au/mSiO2 with better performance was obtained by seed-growth route in a biphasic medium. This synthesis provides particles with 100 nm diameter and pore distribution between 5 and 13 nm, determined by N2 adsorption, which remains unchanged up to 5 days in simulated biological media (PBS). These particles retain proteins (BSA, RFP) and enzymes (HRP) more efficiently than particles with 3 nm pores or non-porous particles, confirming that proteins lodge in the pores. In the case of enzymes, the activity is comparable to that of the enzyme in solution. These findings represent a promising stride towards the advancement of a NV for controlled delivery and release of enzymes into cells.

Designing Hybrid Functional Aerogels forEnvironmental Applications

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The world today confronts numerous environmental challenges that require the creation of functional materials with exceptional (photo)catalytic activity and superior adsorption capacity. Inorganic nanomaterials exhibit excellent performance in removing contaminants and adsorbing metals due to their unique electronic and surface properties. However, using nanomaterials in particulate form for water treatment applications is impractical, considering the related economic, energy, and operational difficulties associated with material recovery [1]. In this presentation, we will discuss our recent studies [2,3] exploring bacterial nanocellulose-based aerogels as a porous, mechanically-stable and flexible support for inorganic nanostructures in order to design hybrid aerogel membranes for in-flow photo-assisted water treatment. Combining sol-gel, hydrothermal and supercritical drying techniques, we achieved controlled deposition of mesoporous metal sulphide/oxide layers on bacterial cellulose (BC) nanofibrils, thus obtaining lightweight hybrid organic-inorganic aerogel membranes. The prepared hybrid aerogels were employed in a specifically designed membrane photoreactor for the photo-assisted removal of organic and inorganic contaminants, such as dyes and heavy metals. Correlation between synthesis, material characterization, structural, and photocatalytic properties has yielded significant insights into designing functional materials for in-flow photocatalytic water purification. The optimized hybrid aerogel membranes exhibit promising performance for application in in-flow photo-assisted water treatment, representing a significant advancement in the use of free-standing aerogel membranes for photocatalytic applications in liquid media.

References

1-Ullah, S. et al *Photochem. Photobiol. Sci.*, 22, 219-240 (2023)
2-Ferreira-Neto, E.P. et al*ACS Appl. Mater. Interfaces*12, 41627-41643 (2020)
3- Ferreira-Neto, E.P. et al *ACS Appl. Mater. Interface*s, In press, (2023)

Development of a thermoresponsive nanobiohybrid material to control enzymatic activity

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Smart materials are mainly responsive materials that have one or more properties that can be controlled by external stimuli. They became a key research topic because of their versatility to be applicable in many areas, such as controlled release, and sensors. In numerous studies, these materials can be combined with other materials or molecules, forming hybrid materials or composites aiming to create or improve some properties [1]. For this work, the thermoresponsive polymer poly(N-isopropylacrylamide) (PNIPAm) was combined with gold nanoparticles (AuNP) previously functionalized with the Candida antarctica (CALB) enzyme. The aim is to show that PNIPAm can be used to modulate the enzymatic reaction by variation of temperature. Even following different synthesis protocols, in which the addition of the enzyme occurred in different steps [2], the structures formed were stable and presented similar characteristics. Transmission electron microscopy (TEM) images indicated that the hybrid material has been obtained. The enzymatic activity showed that the immobilization of CALB still maintains good activity, while the reaction with AuNPs alone behaved similarly to autohydrolysis. The ability to control enzymatic reactions by temperature has significant applications in various areas, such as biocatalysis, and drug delivery systems.

Acknowledgments: CAPES/PROEX, CNPq, DQ/UFPR, CME-UFPR

References:

[1] Bouhfid, R.; Qaiss, A. K.; Jawaid, M. (ed.) Polymer Nanocomposite-Based Smart Materials: From Synthesis to Application. Duxford: Woodhead Publishing, 2020.

[2] Barros, H. R.; Santos, M. C.; Barbosa, L. R. S.; Piovan, L.; Riegel-Vidotti, I. C. J. Braz. Chem. Soc. 30, 2231 (2019).

Laser-Driven, Local Implementation of Polymer Functionalities into Mesoporous Thin Films Enabling Selective Ionic Transport in 3D

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Their aptitude for transport modulation of ionic species renders mesoporous ceramic polymer hybrid materials integral components of novel energy management, sensing, and molecular sieving strategies. Nature's unrivaled precision in ion transport evolves from pore architectures refined to the nanoscale and structurally adapted to the demands of the traversing ions. In search for a concept, transferring the structuring precision to artificial mesopores, the plasmonic field of Au nanoparticles, entrapped in mesoporous silica double layers at a defined z position, was employed as directing unit for nanolocal placement of pH responsive polymers. Following a visible light-induced, oxygen-tolerant (PET)-RAFT protocol, the localized surface plasmon-induced polymerization was identified in IR, TGA, and TEM.[1] With laser-driven polymer writing we present an additional robust tool for locally precise implementation of polymer functionalities into mesopores.[2] We further work on gaining deepened understanding of how fine-tuning of reaction parameters and selection of polymerization reaction affords local control over polymer placement. The technique is shown to be a gateway to polymer barriers, i.e., linear arrays of polymer spots down to 7 μm in diameter, in x/y direction,[2] thus paving the way toward control over ionic motion in 3D.

References:

[1] D. John, M. Stanzel, A. Andrieu-Brunsen, Adv. Funct. Mater. 2021, 31, 2009732.
[2] C. Förster, R. Lehn, A. Andrieu-Brunsen, Small 2023, 2207762.

Naphthalenediimides(bis)urea supramolecular synthons to build up molecular (1D)-fibers

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Naphthalene diimides (NDIs) are excellent compounds to form supramolecular systems properly due their planar structure, p-acidity, well-defined photophysics, and relatively easy synthesis [1]. In the present study, NDIs-(bis)ureas were prepared with donor and acceptor groups in order to investigate aggregation in solution and figure out how these groups may interfere with the molecular packing, forming either segregated solids or blended ones. UV-vis and fluorescence measures pointed to electron donor/acceptor driven-interaction of the NDI-(bis)ureas, and FT-IR, NMR, PXRD and SEM analysis indicated the formation of blended fibers. Therefore, such outcomes emphasise the importance of the stereoelectronic groups for building molecular blends and trigger intermolecular interaction. Moreover, an overview of the concepts and perspectives of this fascinating area belonging to crystal engineering, in which this study is embedded, will be highlighted.

Acknowledgements: FAPESP regular grant n° 21/01509-0 and BPE-FAPESP 22/06346-5; Prof. Jonathan W. Steed of the Durham University-UK who is the main collaborator of thisstudy.

References
Bhosale, S. V., Al Kobaisi, M., Jadhav, R. W., Morajkar, P. P., Jones, L. A., George, S. Naphthalene diimides: perspectives and promise. Chemical Society Reviews, 50(17), 9845-9998 (2021).

TPDH Nanotube: structural, mechanical and electronic properties.

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Carbon nanotubes (CNTs) are highly sought after due to their unique mechanical, optical and electronic properties. They can be visualized as cylindrical tubes formed by rolling two-dimensional sheets of graphene, where the electronic properties of the tubes are dependent on their chirality and diameter. Although graphene is the most used structural base, it is theoretically possible to create NTs using other 2D carbon allotropes [1]. Some of the most widely studied allotropes are Graphenylene, Penta-graphene, HOP-graphene, TPDH-graphene, and others, which consist of networks of linear chain polygons ranging from tetragons to dodecagons [1]. TPDH-graphene, one of the last carbon allotrope, is composed of sp2 carbon rings with 4, 5, 12, and 6 atoms. It exhibits both thermal and dynamic stability, anisotropic elastic properties, and metallic behavior that can be altered by hydrogenation of the tetragonal rings [2,3]. In this work, we explore the structure, mechanical, and electronic properties of TPDH-NTs with chirality (n,0),(n,1), (n,n) for n 4, using density functional theory (DFT) and molecular dynamics (MD) calculations. Our preliminare results indicate that TPDH-NT (2,1) exhibits semiconductor behavior with a small indirect band gap, while other TPDH-NTs show metallic behavior. We observe the structural stability of TPDH-NT via MD at (T=300K), and the stress-strain profiles calculated by DFT and MD show a good correlation, coinciding at the fracture limit of the TPDH-NTs. These preliminary results suggest that TPDH-NTs possess distinct mechanical and electronic properties compared to TPDH-2D, especially TPDH-NT with chirality (2,1), which exhibits semiconductor behavior.

References:
[1] Susmita Jana, et al. Journal of Physics: Condensed Matter, 34, 053001 (2022).
[2] Caique C Oliveira, et al. Physical Chemistry Chemical Physics, (2023).
[3] Debaprem Bhattacharya, et al. Physica E: Low-Dimensional Systems and Nanostructures, 127, 114569 (2021).

Characterization of the Thermodynamic and Kinetic Properties of Thermosensitive Composites for Controlled Drug Release

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Smart polymers, known as stimuli-responsive polymers, are a class of materials that exhibit dynamic and reversible changes in their properties in response to external stimuli. Among them, the thermosensitive ones stand out for their ability to phase transition in response to temperature changes, which can result in changes in solubility, swelling and other physical properties and can be used in drug delivery, sensors and tissue engineering as this change occurs in a controlled and predictable way. However, the narrow transition temperature range of some materials is a challenge that implies modifying this phenomenon. The modification of this transition, and consequently of the properties of the composites, is done by adding nanoparticles to the polymeric matrix in order to change the temperature, width, and hysteresis of the polymer, providing a tunable and controllable phase transition behavior. The objective of this work was to study the influence of the addition of SiO_2 nanoparticles to the poly(N-vinylcaprolactam) matrix on its thermodynamic and kinetic properties, aiming at optimizing the transition profile for using the material in controlled drug delivery system. An in situ, polymerization of PNVCL was carried out in the presence of 20nm SiO_2 nanoparticles and the composites were subjected to optical, spectroscopic and physicochemical characterizations. The results showed a strong influence of the nanomaterial on the molecular mass and transition temperature of the PNVCL chains. It was observed that increasing NPs loading increased Mw, decreased LCST, and favored smoother transitions. Also, NPs increased initial polymerization speed but decreased conversion rates. Thus, this change in temperature and transition width allows the material to be used as a drug release vehicle in a satisfactory manner.

AKNOWLEDGEMENTS

This work was partly funded by FAPESP(2013/07296-2) and (2022/10016-0), CNPq (309711/2019-3).

Developmente of a E-nose sensor array for detection of VOCs (Human Breath Biomarker) using GO-CoFeO4 nanocomposites.

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Graphene oxide-cobalt ferrite (GO-CoFeO4) nanocomposites have been recently explored as a promising sensors for the detection of breath biomarkers. The GO-CoFeO4 possess high sensitivity and selectivity towards various volatile organic compounds (VOCs) that are present in exhaled breath, which can be indicative of specific diseases or metabolic disorders. The cobalt ferrite nanoparticles improve the electrical properties of GO leading toa change in electrical conductivity of the nanocomposite thick films and better sensivity. A serie of nanocomposite were rationally sythesized leading to successfully detection of several breath biomarkers, including acetone, ammonia, formadehyde and alcohol. The PCA analysis showed specifics regions for each biomarker. The fabrication of GO-CoFeO4 nanocomposites is simple and cost-effective, making them a promising candidate for the development of low-cost and portable breath biomarker sensors for early disease diagnosi

Development of Hybrid Siloxane-Polyether Nanocomposites containing Superparamagnetic Nanoparticles for Future Applications in Drug Releaseby Magnetic Hyperthermia

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In this work siloxane-polyether (PEO or PPO) hybrid nanocomposites containing □-Fe_2O_3 nanoparticles functionalized with Triethyleneglycol (TEG) have been prepared by Sol-Gel process, aiming future applications of controlled drug-delivery by magnetic hyperthermia. The magnetic particles were prepared by solvothermal method in a microwave reactor, and dispersed in the hybrid sol during the hybrids synthesis. X-ray Diffraction measurements (XRD) showed the characteristic crystalline phases expected for □-Fe_2O_3 superparamagnetic nanoparticles, whereas Dynamic Light Scattering (DLS) and Transmission Electron Microscopy (TEM) revealed that particles are monodispersed, with ranging from 5 to 15 nm, depending on synthesis condition. Magnetic analyses showed that, at room temperature, the particles exhibit a superparamagnetic behavior and that the dipolar interaction between the nanoparticles is weak. Magnetic hyperthermia measurements have been carried out with the synthed particles, to determine the optimal conditions (amplitude and frequency of the magnetic field) that lead to the optimum heat generation. The first synthesis of the hybrid nanocomposites evidenced the possibility to incorporate significant amounts of magnetic particles in Siloxane-PEO hybrid matrix, due to the hydrophilic nature of TEG and PEO, while dispersion of the particles in the more hydrophobic Siloxane-PPO matrix is inhibited. These preliminary results are promising for the future development of biocompatible, transparent and flexible hybrid films presenting high stability in biological media, which may be employed for drug release in human skin by magnetic hyperthermia.

Acknowledgements:
Authors gratefully acknowledge the LaMAR/Universidade Federal Fluminense for electron microscopy facilities and the financial support of Brazilian agencies FAPERJ (Nanotechnology Research Redes Program in the state of RJ), CAPES and CNPq.

Dip Pen Nanolithography of Gold and Magnetite Nanoparticles

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Nanoparticles offer unique physical and chemical properties. Dip pen nanolithography of nanoparticles enables versatile patterning and nanofabrication with potential application in electronics and sensing, but is not well studied yet. Herein, the patterned deposition of various nanoparticles onto unmodified silicon substrates is presented.[1] It is shown that aqueous solutions of hydrophilic citrate and cyclodextrin functionalized gold nanoparticles as well as poly(acrylic) acid decorated magnetite nanoparticles are feasible for writing nanostructures. Both smaller and larger nanoparticles can be patterned. Hydrophobic oleylamine or n-dodecylamine capped gold nanoparticles and oleic acid decorated magnetite nanoparticles are deposited from toluene. Tip loading is carried out by dip-coating, and writing succeeds fast within 0.1 s. Also, coating with longer tip dwell times, at different relative humidity and varying frequency are studied for deposition of nanoparticle clusters. The resulting feature is between 300 and 1780 nm as determined by scanning electron microscopy. Atomic force microscopy confirms that the heights of the deposited structures correspond to a single or double layer of nanoparticles. Higher writing speeds lead to smaller line thicknesses, offering possibilities to more complex structures. Dip pen nanolithography can hence be used to pattern nanoparticles on silicon substrates independent of the surface chemistry.

Acknowledgements:
This work was funded by the Deutsche Forschungsgemeinschaft (DFG CRC 1459 Intelligent Matter - Project-ID 433682494 and INST 211/719-1 FUGG for TEM instrumentation). The authors thank the Münster Nanofabrication Facility for providing instruments for deposition and analysis. The authors were grateful to Uwe Bog (n.able) for support with the molecular printer.

References:
[1] L. Schlichter, F. Bosse, B. J. Tyler, H. F. Arlinghaus, B. J. Ravoo, Small 2023, 19, 2208069.

Enhancement of the red emission of Eu3+-complexesby plasmonic nanostructures for cell imaging application

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Metal-Enhanced Luminescence (MEL) is an effect explored to enhance the emission intensity and maximize other photophysical parameters of lanthanide ions (Ln3+) that are nearby plasmonic nanostructures such as metallic gold or silver nanoparticles [1]. Here, the objective was to evaluate the luminescent and biological properties of plasmonic nanostructures (Ag@SiO2) functionalized with luminescent europium complexes (Eu3+-complex). The development of such structures was performed in several reaction steps. First, citrate-stabilized 30 nm-AgNP were covered with silica thickness of 37 or 53 nm, resulting in the Ag@SiO2 structures, which were later functionalized with APTES (3-aminopropyltriethoxysilane) and, finally, had their amino groups reacted with the epoxide group present in the previously synthed complex [Eu(TTA)3ephen], TTA = 2-thenoyltrifluoroacetone, and ephen = 5,6-Epoxy-5,6-dihydro-[1,10] phenanthroline. Photoluminescence analysis showed that the presence of AgNP enhanced the emission of the anchored complexes by a factor of 17 times when compared to the emission of the materials used as blank (50 and 170 nm silver-free silica nanoparticles functionalized with the same Eu-complexes). Cytotoxicity assays on liver cancer cells, Huh 7.5, and confocal microscopy measurements showed that the plasmonic structures were non-toxic at 25 mg/mL and were internalized by cells, preserving the Eu3+ red-emission and the cell structure.

Acknowledgments:
CNPq, CAPES, and FAPESP (19/26103-7).

References:
[1] Liu, J.; Wang, Q.; Sang, X.; et al. Nanomaterials. 11, 1037, (2021).

Epoxy-silica anticorrosive coatings modified with Ce (III) and Ce (IV) ions with self-healing properties

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Coatings with an efficient anticorrosive barrier are essential for the protection of metal surfaces, however over time the coated substrate becomes susceptible to localized corrosive action and physical damage, such as cracks and scratches, reducing its protection efficiency. To ensure long-term protection against corrosion, coating systems must combinea dense and effective passive barrier against the permeation of oxidizing species and active protection of surfaces through a self-healing capacity after failures. The self-healing property can be achieved with the addition of self-healing agents, such as Ce(IV) and Ce(III) ions. In this context, hybrid epoxy-silica coatings were modified with the incorporation of cerium salts (Ce(NO3)3.6H2O), (NH4)2Ce(NO3)6) varying the molar concentration between 0.5 and 5.0 % of Ce(III) and Ce(IV), in relation to the total silica present in the hybrid. The epoxy-silica hybrids were prepared from the curing of poly(bisphenol-A-co-epichlorohydrin) (DGBEA), (3-glycidoxypropyl)trimethoxysilane (GPTMS) and diethyltriamine (DETA), combined with hydrolysis and condensation reactions of tetraethoxysilane (TEOS) in aqueous acidified ethanol. Films with a thickness of about 15 μm were deposited by the dip-coating method on aluminum alloy yielding and adhesion strength of 20 MPa. To evaluate the effect of the addition of Ce(III) and Ce (IV) ions on the nanostructure formation, SAXS, FTIR, TG and contact angle measurements were used. The results obtained by electrochemical impedance spectroscopy in NaCl solution showed a high corrosion resistance of the coatings with low-frequency impedance (4 mHz) of about 100MΩcm2, that is, 5 orders of magnitude higher than the bare substrate. The self-healing effect was evaluated for scratched coatings after immersion in saline solution. After 10 days it was possible to observe a recovery of the impedance modulus, evidencing the self-healing ability of the coatings.

Hybrid urethane films based on PDMS and OGPOSS: Synthesis via CO2 fixation and evaluation of its antimicrobial activity.

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In this study, we have developed potential antimicrobial polymeric films for possible applications as antimicrobial metal coatings. The precursors were prepared by the reaction between carbon dioxide and polyepoxides, in order to obtain cyclic bis- and octa-carbonate (CC''s). Thus, these CC''s were reacted by aminolysis with isophorenediamine and an aminopropytriethoxysilane, leading to the formation of non-isocyanate (poly)urethanes (NIPU''s) derived from poly(dimethylsiloxane) (PDMS) and octaglycidyldimethylsilyl polyhedral oligomeric silsesquioxane (OGPOSS). Finally, self-supported hybrid films of these NIPU''s were obtained through the sol-gel process, catalyzed by phosphotungstic (PWA) or phosphoric (H3PO4) acid. The precursor CC' s and the hybrid films were characterized by the techniques of Energy Dispersive X-ray Fluorescence Spectroscopy (EDXRF), Fourier Transform Mid-Infrared Absorption Spectroscopy (FTIR), Raman Spectroscopy and Nuclear Magnetic Resonance (NMR). In addition, the films were preliminarily tested for bactericidal activity for Staphyloccocus aureus. The acid incorporation into the urethanes improved the antimicrobial activity from bacteriostatic to bactericidal. Higher concentrations of CCOGPOSS also implied an improvement in antimicrobial activity.

Acknowledgments:
This work was supported by the research funding agencies CAPES and FAPESP. The authors would like to thank the postgraduate program in Materials Science and Engineering from EESC-USP and all the collaborators in the group of hybrid and inorganic materials chemistry (GQMatHI) from IQSC-USP.

In-situ polystyrene/BaSO4 nanocomposites via multiple sequencial microemulsions using phase inversion temperature (PIT) as emulsification low-energy technique

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Polymer/inorganic nanocomposites have received considerable attention of academic and industrial research as the incorporation of nanofillers within a polymer matrix often leads to materials with enhanced mechanical, thermal, or barrier properties, even at low inorganic contents [1]. However, obtaining these nanoparticles as well as stabilizing them proves to be a particularly challenging task, mainly via heterophase polymerization in aqueous media. Several works in the literature have been reported for miniemulsion polymerization as an alternative to overcome these drawbacks, however with the help of poorly available and expensive reagents, or even using specific equipment with clear limitations of scale-up to industrial end-use, what severely limited their adoption by the main targeted end-applications [2]. In this context, the sequential processing of multiple reactions in mini emulsion, using conventional reactors, presents itself as an interesting alternative to achieve the objectives, both for the generation of inorganic nanoparticles and the respective hybrid latexes. This work aims to carry out a model study to enable both guided and in-situ synthesis of inorganic nanoparticles of BaSO4 to act as pigments, as well as that of hybrid latices of polystyrene/BaSO4 as seed for acrylic film-forming agents, base materials for water-based decorative paint formulations, obtained through the sequential processing of multiple (mini)emulsions using a combination of low-energy technique with temperature induced emulsification (PIT) and catastrophic phase inversions, using technical grade nonionics and anionic as surfactants in this two-step radical polymerization approach.

[1] A. Bhat et al, Smart polymeric nanocomposites: synthesis and applications, Nanotechnology Reviews (2021). doi: 10.1515/ntrev-2021-0018

[2] J.M Asua, Challenges for industrialization of miniemulsion polymerization. Progress in Polymer Science (2014). doi: 10.1016/j.progpolymsci.2014.02.009

Lanthanide doped luminescent nanoparticles for application in chemical and biological sensors

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Upconversion nanoparticles (UCNPs) have high versatility of application and are capable of emitting light with a wavelength shorter than the excitation, converting low-energy light into high-energy light. UCNPs have been used in various applications, but it is probably in the field of detection that they best demonstrate their potential. Thanks to the possibility of controlling cytotoxicity, high photo-stability, large anti-Stokes shifts, long lifetimes, and reduced background noise, which significantly increases the limit of detection (LOD), the emerging field of UCNP-based sensors is rapidly evolving through various approaches. We designed matrices of NaYF4 and NaGdF4 varying the pairs of doping ions Yb3+/Er3+ and Yb3+/Tm3+. With excitation at 980 nm, Er3+ presents emission peaks around 520, 540 and 650 nm and Tm3+ around 350, 450, 480 and 650 nm. Since UCNPs allow the use of low-energy excitation wavelengths, it is possible to achieve ideal energy excitation windows in the biological sensors, gaining particular prominence in this field. Also, within the scope of sensors and lanthanide-doped nanomaterials, we studied Nd3+-doped nanoparticles (Nd-NPs). Exciting these nanoparticles at 808 nm, emission peaks arround 880 and 1060 nm were analized to be used as parameters for temperature sensors in nanothermometry. Thus, in this work we optimized the synthesis by high temperature coprecipitation and characterize NPs consisting of different matrices and doping ions. For the UCNPs, the exchange of Y3+ for Gd3+, with both doping ions, showed a noticeable diameter reduction of approximately 3,5 - 4,6x with maintenance of luminescent properties and crystalline phase. On the other hand, the thermometric characterization of Nd-NPs are still in progress but already shows promising luminescent results. In this way, the poduced materials present potencial to be used in different multifunctional platforms in order to achieve the desired applications.

Nanostructured coatings based on copper/functionalized carbon nanotubes doped withiodine (Cu/NTC-f-i)

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New nanostructured conductors developed from the coating of aluminum wires Al 1350 by copper/carbon nanotubes functionalized and doped by iodine (Cu/NTC-f.i), using the process of electrophoretic deposition in copper sulfate solution, under electric currents of 1.2 to 1.8 A, have been investigated for their electrical properties. The Al@Cu/NTC-f.i conductors were characterized by scanning electron microscopy; X-ray diffractio Raman spectroscopy, in addition to electrical measurements, at room temperature and under heating, via a Kelvin bridge. The results show an increase of approximately 18% of the IACS with good stability under heating compared to the commercial aluminum conductor Al 1350, due to the intrinsic properties of the functionalized CNTs and the doping effect of the iodine, confirmed by Raman spectroscopy with redshifts effects in the sub- G-band bands, in addition to ACD calculation and FWHM data. The characteristic morphology of the Cu/CNT-f thin films showed pyramidal structures, agglomerates, copper structures involving the CNTs, as well as CNTs homogeneously electrodeposited on the aluminum substrate withgood crystallinity, cultivated mainly in the directions (311) and (111). This Cu/NTC-f.i electrodeposition method on the surface of metallic wires shows promise for the productionof nanostructured metallic conductors with high ampacity, thermal stability, low density andhigh performance, applied in electric energy transmission lines. for application in power transmission lines.

SiO2/graphene oxide nanocomposite obtained from rice husk pyrolysis

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Rice husk contains cellulose, hemicellulose, some inorganic materials such as silica and a considerable percentage of lignin, the second most abundant natural polymer in nature [1]. Previous studies have shown that the crystallinity of carbon materials such as lignin can be improved by exposure to high temperatures, typically greater than 700°C, in an inert atmosphere [2,3]. In this work, the thermoconversion of rice husks was carried out to produce a SiO2/graphene oxide nanocomposite, where the lignin nanocarbon is a dispersed phase in a silica matrix. The silica is present in the basal plane of the rice husk, and after lignin thermoconversion, the produced nanocomposite was incorporated into cementitious pastes, providing improved resistance to compression. The chemical structure of the rice husk was modified by a controlled routine based on sequential irradiation with ultraviolet light, hydrothermal carbonization in an autoclave, vacuum degassing, pyrolysis and mechanical exfoliation. The SiO2-GO nanocomposite was characterized by X-ray diffraction, Fourier-transformed infrared and Raman spectroscopies and X-ray photoelectron spectroscopy. The thermal treatment performed was not sufficient to degrade the silica, allowing only the transformation of the lignin present in the rice husk in a nanoporous carbon structure composed of sp2 hybridized lignin structure with oxygenated functional groups attached to this structure. The Raman and infrared spectra revealed the presence of two phases in the structure, one related to the carbon crystallite grains and the other related to silicates, reinforcing the idea of a nanocomposite between the two phases.

Acknowledgements: CNPq, Faperj and Fundep.

References:
[1] LEE, Changgu et al., Science, v. 321, p. 385-388, 2008.
[2] RIBEIRO-SOARES, J. et al., Carbon, v. 95, p. 646-652, 2015.
[3] SOUZA, Milton. et al., FAPESP, v. 58 , p. 56 - 58, 2000.

Sound crystallization of anthranilic acid polymorphs

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Anthranilic acid (AA) crystals are known in three polymorphic forms, form I (AMBAC007), form II (AMBAC005), and form III (AMBAC008). Phase I is triboluminescent, that is, ability to emit light when it is under pressure or sudden extension. Further, phase I has unit cells with polar space groups, while phases II and III are nonpolar [1]. We have developed a mechanical-acoustic system to crystallize separately AA polymorphs. The procedure consisted in preparing a supersaturated solution of AA (solute) in distilled H2O (solvent) in a recipient (Teflon or stainless steel), then, it was put on plate under sound waves with certain low frequency and intensity. Once crystals solidified out, they were filtered off and dried in an oven at 70°C for 24 hours. Solid phases were identified by PXRD and irradiation underUV light (l = 365 nm). Depending on the acoustic setup it was observed both concomitant polymorphs and pure (single phase) crystals. So far, it is the first description of crystallizing different AA solid forms from the same solvent. Moreover, such unprecedent mechanical-acoustic crystallization is rather promising for both supramolecular chemistry area and industrial crystallization operations.

Acknowledgements:

Grant nº 2021/01509-0, Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP).

References:

[1] Gordon E. Hardy, William C. Kaska, B. P. Chandra, and Jeffrey I. Zink. Triboluminescence-Structure Relationships in Polymorphs of Hexaphenylcarbodiphosphorane and Anthranilic Acid, Molecular Crystals, and Salts. J. Am. Chem. Soc., 103, 1074-1079, (1981)

Sounds waves for generating polymorphic crystals ofROY

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The new mechanical acoustic method for generating polymorphic crystals is a revolutionary approach to crystallization. The process occurs out of equilibrium, using a supersaturated solution of 5-methyl-2-[(2-nitrophenyl)amino]-3-thienylacetonitrile, or ROY (Red, Orange and Yellow), a substance with approximately 12 different polymorphs cataloged [1] in distinct solvents such as isopropyl alcohol and ethanol. The solution is placed in a container (teflon or stainless steel) and subjected to low-frequency, low-intensity sound waves, causing vibration on a plate. By varying the frequency and intensity of the sound waves, different or concurrent polymorphs can be generated, with variations in their growth axes and consequently resulting in distinct colors. This method allows for the reproduction of polymorphs based on the specific variations of the frequency and intensity of the sound waves applied. Once crystals are formed, the solution is filtered and dried in a 70°C oven for24 hours. Solid phases are identified by PXRD. Therefore, the use of this mechanical- acoustic method can greatly innovate industrial and supramolecular crystallization.

Acknowledgements:
FAPESP - Grant nº021/01509-0

References:
[1]. Yu, L. (2010). Polymorphism in molecular solids: an extraordinary system of red, orange, and yellow crystals. Accounts of chemical research, 43(9), 1257-1266

Synthesis and characterization of a pH-sensitive luminescent hybrid system for theranostic applications

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Medical advances have improved the survival of cancer patients. Nevertheless, the lack of distinction between normal and tumor cells causes several side effects. Therefore, the scientific community has explored the development of pH-responsive polymeric systems, that can promote drug delivery to cancer cells. Furthermore, these polymers can be associated with materials such as mesoporous silica, hydroxyapatite, and rare earth elements, to obtain a multifunctional system that can act in therapy and diagnosis simultaneously, known as theranostic materials. Mesoporous silica has exciting properties as the high surface area and hydroxyapatite have been widely studied to aid bone regeneration. When associated with rare earth elements, such as europium, and gadolinium, these materials become the potential for imaging diagnosis. Therefore, the objective of the present work is to obtain a hybrid system responsive to pH based on silica and hydroxyapatite with photoluminescent and magnetic properties. The materials were characterized by FTIR, XRD, SEM, TGA, XPS, superficial analyses, photoluminescence, and zeta potential techniques. Incorporation and release studies of the antitumor drug doxorubicin were carried out to evaluate the potential use of these systems in the targeted delivery of drugs. The results evidenced the luminescent and magnetic properties of the materials and showed characteristics suitable for application in pH-sensitive drug delivery.
Acknowledgments: CAPES, CNPq, FAPEMIG, and CDTN.

References:

[1] Bhattacharya, S., Prajapati, B. G. Singh, S. Journal P. Crit. Rev. Oncol. / Hematol. 103961 (2023)

[2] Apostolos, R. C. R., Cipreste, M. F., de Sousa, R. G. de Sousa, E. M. B. Multifunctional hybrid nanosystems based on mesoporous silica and hydroxyapatite nanoparticles applied as potential nanocarriers for theranostic applications. J. Nanoparticle Res. 22, (2020).

SYNTHESIS AND CHARACTERIZATION OF HYBRID FILMS BASED ON URETHANESIL MATRIX AND PHOTOACTIVE SILICA AEROGELS@ANATASE@PRUSSIAN BLUE NANOCOMPOSITE

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The field of photoactive device design has received significant attention in recent years due to its impact on technological advancements. One promising approach to enhance the performance of these systems is to prepare semiconductor oxide nanocomposites with metals or metal complexes. This can lead to improvements in (photo)catalytic activity, stability, solar energy conversion, and the addition of new properties. Core@shell nanocomposites are particularly interesting due to their ability to tune structural and electronic properties by modifying the composition and physicochemical properties of their core and shell components [1].

In this study, we explore the modification of titanium oxide-based photocatalysts with hexacyanometallate Ferric Ferrocyanide, also known as Prussian Blue (PB). Titania-silica aerogels decorated with PB (ae-SiO_2@TiO_2@PB) were synthed combining sol-gel and photodeposition methods techniques. We studied the photochromic properties of titania silica aerogel decorated with PB dispersed on thin films based on hydroxiurethane/polydimethylsiloxane (PDMS) urethanesil type hybrid [2]. Prepared hybrid films show reversible photochromic behavior based on photo-induced reduction of PB layer by anatase nanocrystals under UV-light. Our results suggest that semiconductor@PB core@shell nanocomposites have promising potential for the design of photoactive devices in a variety of applications, including photochromic thin films, solar cells, and photocatalytic surfaces.

[1] Ferreira Neto, Elias Paiva et al. Prussian blue as a co-catalyst for enhanced Cr (VI) photocatalytic reduction promoted by titania-based nanoparticles and aerogels. New J. Chem.. , p. 10217-10231. abr. 2021

[2] Aguiar KR, Santos et al: Efficient green synthesis of bis(cyclic carbonate)poly(dimethylsiloxane) derivative using CO_2 addition:anovel precursorfor synthesis of urethanes. RSC Adv 2014, 4(46):24334.

Acknowledgments:CAPES and FAPESP

Transmission of acoustic waves and logic operations onY-shaped nonlinearly coupled harmonic chains

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We investigate the propagation of acoustic waves on Y-shaped coupled harmonic mass-spring chains and discuss the possible realization of logic functions. Non-linear contributions to the coupling among the three semi-infinite chains are considered. The stationary scattering theory is used to obtain exact expressions for the transmission spectrum on the output channel for the cases of incident waves coming from a single or both input channels. Special features related to the nonlinear character of the coupling are explored including bistable behavior and self-induced transparence. Considering an amplitude modulated protocol with a threshold transmission level, we explore the possible realization of logic functions. We determine the spectral regions for the realization of different logic operations as a function of the wavenumber of the incident normal modes and the relative phase difference between them.

Acknowledge: The authors acknowledge the partial support by CNPq and FAPEAL

C-Multifunctional Polymer Blends and Composites

On a layered composite coating with piezoresistivity and mechanoluminescence properties for wearable sensors

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The development and validation of a multifunctional composite material with visible sensing behavior remains still a challenge. However, graphene and its derivatives, represent a valid tool to design responsive materials, through a fine controlling of their spatial distribution and interfacial interactions with polymeric phases [1, 2]. Here we present a smart fabric with piezoresistivity and mechanoluminescence ability based on a layered structure made of graphene-modified conductive cotton fabric and $SrAl_2O_4:Eu^{2+}$, Dy^{3+}/polyurethane foamed coating. The presence of the layered structure endows the flexible composite fabric with a controllable positive/negative piezoresistivity (gauge factor from -2.5 to 17) and a bright mechanoluminescence ability that can be used in the darkness to detect specific deformations, without additional batteries. Three-dimensional chromaticity diagrams have also been developed as advanced tools to quantitatively assess the luminescence, whose lightness during the bending cycles is highly repeatable. The multifunctional layered composite coating provides multiple output signals which can be accurately recognized and assigned to independent triggering events, opening up to new opportunities in advanced wearable devices based on commercial fabrics [3].

Acknowledgements:

This work was partially financed by the European Union Next-Generation EU, Italian National Recovery and Resilience Plan (PNRR)-Mission 4, Component 2, Investment 1.3-Research and Innovation Programme “3A-ITALY”-PE00000004-CUP B73C22001270006.

References:

[1] M. Salzano de Luna et al., Progress in Polymer Science 89, 213-249, (2019)

[2] Y. Zhan et al., Composites Science and Technology, 205, 108689 (2021),

[3] Jize Liu et al., Composites Science and Technology 236, 109993(2023)

Towards smart circular manufacturing strategies - a materials revalorization perspective

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Humanity faces dramatic challenges, as humanity is exhausting nature’s budget progressively each year. At the same time, waste is growing exponentially; consumption reduction, reusing and recycling are no longer options but vital for sustainable human activities on earth. The concept of a circular economy aims to address these issues by closing the material loop across the full supply chain. Yet, significant barriers prevent its full adoption. Major ones being lack of information on the product life cycles and shortage of advanced technologies for remanufacturing and novel reverse supply chain strategies for material revalorization. Research on taking advantage of smart technologies to unlock the full potential of circular manufacturing approaches is just starting.

Besides the discussion on intelligent CLSC approaches, ongoing research activities on sustainable material development and a framework for study on improving performance of circular manufacturing will be presented.

3D printing of electrically conductive polymernanocomposites for fuel cell applications

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Extrinsic electrically conductive polymer nanocomposites can be produced by adding conductive nanoparticles to a polymer matrix. In particular, carbon-based nanomaterials, such as graphene, carbon nanotubes and carbon black, are effective to increase the electrical conductivity of polymers without changing the weight of the material significantly [1]. Fuel cells can take advantage of such materials, especially when related to automotive applications. Fuel cell parts such as bipolar plates can be made lighter than usual if produced from conductive polymer nanocomposites [2]. Additive manufacturing can also be an important ally in the production of such devices, as the design of the parts can be promptly adjusted and optimized in an iterative manner [3]. In this work, different types of thermoplastics were combined with carbon-based nanomaterials, to produce a series of electrically conductive nanocomposites. The samples were prepared by twin screw extrusion and solvent-assisted techniques, and their electrical, mechanical and rheological properties were characterized. 3D printing filaments were subsequently produced and used to print test samples and prototypes of bipolar plates for fuel cell applications. High electrical conductivity was reached by a series of samples, depending on their composition, type of conductive filler and processing technique used. The direction of 3D printing also affected the final conductivity and overall performance of the printed samples.

Acknowledgements:
EMBRAPII, Volkswagen do Brasil, Skintech Tecnologia, FINEP, IPEN

References

[1] K. Ke et al., Carbon, v. 173, p. 1020 (2021)
[2] B. Hu et al., Energy, v. 251, p. 123982 (2022)
[3] S. Pejak et al. Emerging research in conductive materials for fused filament fabrication (FFF): A critical review. Advanced Engineering Materials, v. 24, p. 2101476 (2022)

A NanoCodec: 3D Printed Nanostructured Quick Response Code with Physically Unclonable Function Based on Carbon Nanotubes

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Due to the Industry 4.0, factories are increasingly dependent on the Internet and communication technologies, which guarantees unprecedented efficiency, but can also lead to vulnerabilities [1]. For this reason, cybersecurity is an increasingly relevant topic, with technologies such as physically unclonable functions (PUFs) and quantum cryptography being good alternatives in this area [2]. For this reason, this work presents the development of a nanocomposite of poly(acrylonitrile-butadiene-styrene) (ABS) and carbon nanotubes (CNTs) for the 3D printing of so-called NanoCodec, which present spectral signatures that can be used as PUFs in quantum cryptography. For this, ABS/CNTs nanocomposite filaments were produced, with 1 and 2 wt% of CNTs, for printing the NanoCodecs in quickresponse code (QRcode) and in circular/square stamps by fused deposition modeling (FDM). Raman spectroscopy was used to characterize the pure materials, both ABS and NTCs, andcompare them with the nanocomposites of ABS/NTCs. This showed an overlap of somebands associated with each material, particularly in the 1580-1620 cm-1 region, where vibrations of both CNTs and ABS carbon-carbon bonds are present, indicating the interaction between them and that the Raman spectrum is affected by the nanocomposite production process, so the random arrangement of CNTs in this material could be used as PUF quantum keys for encryption. Lastly, a mobile app was developed to read the QRcode, encrypt the Raman Spectra of the NanoCodecs, obtained from a Handheld Raman Spectrometer, on a 20-character alphanumeric key to validate the information on a server, which can be used to give trustworthiness on production chains of Industry 4.0.

Acknowledgements:

Thanks to FAPESPA, Finep (nº 01.22.0133.00) and MIDR (n° 10/2020).

References:

[1] V. Alcácer and V. Cruz-Machado, Eng. Sci. and Tech. vol. 22, pp 899-919 (2019)

[2] Moon et al., American Chemical Society vol. 1, no. 7, pp. 1162-1168 (2019)

Development of a Nanoimprinting Process for Antireflective Glass Surfaces

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Antireflective (AR) surfaces are critical developments for electronic displays, solar panels, LED/OLED lighting, and lenses. Biomimetic moth-eye nanostructures offer significant reductions in glass reflection across the visible and near-IR spectrum and are self-cleaning due to their hydrophobic nature. However, current manufacturing methods for AR glass surfaces require clean-room environments and expensive machinery. In order to help the wide-scale adoption of AR glass for solar panels, it is important to further optimize the manufacturing process for these AR surfaces. Nanoimprint lithography (NIL) is a low-cost and high throughput patterning process which is well studied for low transition temperature polymers. Though promising for the large-scale production of AR patterns, high transition temperature glass NIL remains difficult due to the fragility of glass and the precise control required for the high processing temperatures and pressures. This research aims to develop an equipment and process for glass NIL. Ceramic micropatterned moulds were created by Laser-Induced Plasma-Assisted Ablation (LIPAA) and a high temperature testbench compatible with MTS compression machines was developed to replicate mould features. This study will provide finite element analysis (FEA) comparisons to the experimental results to optimize this novel process for high throughput glass imprinting.

Evaluation of physical-mechanical properties ofparticleboards bound with polyethylene terephthalate andcastor oil-based polyurethane resin

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Consumption of natural resources and waste generation has increased significantlyin recent decades. Polyethylene terephthalate - PET is a material widely used for variouspurposes such as in the packaging sector, however, the search for new methods to recycle thismaterial has been significant. Wood, on the other hand, is a natural resource used in theproduction of furniture and structural elements in civil construction, however, its processinggenerates a lot of waste, such as shavings that can serve as an alternative material inreplacement for the raw material for the production of particleboards. In this workparticleboards were produced with pine wood waste, PET particles and polyurethane adhesivederived from castor oil —PUR in the proportions of 50% PET, 50% pine and 10% PUR. Twogroups were studies changing the pressing temperature - 130ºC and 160ºC. Foreach group, 3panels measuring 35x35cm were produced, and their specimens tested for physical andmechanical properties. The results allowed verifying mechanical resistances much higher thanthose required by the Brazilian normative document.

References

[1] R. A. Bispo, et al. Production and evaluation of particleboards made of coconut fibers, pine, and eucalyptus using bicomponent polyurethane—castor oil resin. BioResources, 2022, v. 17, n. 3, pp. 3944-3951.

Influence of sisal fiber on the physical and mechanicalproperties of homogeneous particleboards

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In Brazil, the development of cities has led to the intensification of the exploitationof natural resources. Wood is an example of a versatile resource of great economicimportance to humanity, which is often exploited inconsequentially. An important aspect tobe considered in this study is the use of alternative materials aimed at their use in theproduction of particleboards, consequently, wood particleboards with the addition of sisalfiber were produced and evaluated. In this work, particles of wood from reforestation andsisal fiber were used with nominal density of 0.6 g/cm3, bonded with 10% castor oil-basedpolyurethane resin (PUR) and pressed at a temperature between 150 and 160 °C. Twomixtures were proposed and three panels were produced for each mixture, totaling six panels.For the first mixture, the fibers were distributed close to the surface of the panels and for thesecond, the fibers were distributed in four layers inside the panels. Based on the resultsobtained, it was verified that the addition of sisal fiber resulted in similar performance of thepanels in terms of their physical and mechanical properties and met the criteria proposed byNBR 14810-2/2018.

References

[1] R. A. Bispo, et al. Production and evaluation of particleboards made of coconut fibers, pine, and eucalyptus using bicomponent polyurethane-castor oil resin. BioResources, 2022, v. 17, n. 3, pp. 3944-3951.

Manufacturing of polymer matrix membranes reinforced with nanocellulosic materials for use in wastewater filtration

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Polymeric membranes are applied for wastewater and unhealthy water filtration to contain the action of biological contaminants (bacteria and viruses), wastes from industrial, laboratory, pharmaceutical, and home activities, agriculture, and energy generation [1]. The filtration process can be classified by reverse osmosis, electrodialysis, nanofiltration, ultrafiltration, microfiltration, and distillation membranes [2]. The most common methods of manufacturing polymer matrix membranes are phase inversion, sintering, stretching, track-etching, and electrospinning. There is also the development of thin film nanocomposite membranes, which are developed by in situ interfacial polymerization, dip coating, layer-by-layer assembly, self-assembly, physical, and chemical deposition, chemical grafting, and corona plasma-assisted coating [3]. Nanocomposite materials are good for the reinforcement of polymer matrix membranes due to their high specific area and the fact that they can enhance their capacity to adsorb contaminants of the water, including chemical reagents, such as methylene blue and oxy-halogenates. Many researchers in the world are studying new materials to reach new economic, ecological, and technical advantages, especially nanomaterials. The aim of this study is to compare several processes of filtration, as well as identify and differentiate the application of each one. For this, results from literature were collected about the properties and characteristics of each filtration membrane.

Acknowledgments:
Federal University of Paraná, National Council for the Improvement of Higher Education (CAPES), and National Council for Scientific and Technological Research

References:
[1] M.T.Amin, A.A.Alazba and U.Manzoor, Advances in Materials Science and Engineering vol.2014, 1-24 (2014)
[2] E.O.Ezugbe and S.Rathilal, Membranes vol.10, 1-28 (2020)
[3] M.R.Esfahani et al., Separation and Purification Technology vol.213, 465-499 (2019)

Mechanical Properties of Mixed-Matrix MembranesUsing Coarse-Grained Simulations

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Mixed-matrix membranes (MMMs) are a promising technology to decrease CO_2 emissions by incorporating advanced fillers in their polymer matrices. Carbon nanotubes, for instance, may improve the selectivity/permeability trade-off in traditional polymeric membranes and enhance mechanical stability depending on their density and polymer affinity. Computational simulation modelling is a more efficient and cost-effective way to calculate these properties compared to experimental methods. This study established an effective simulation protocol using Coarse-Grained (CG) molecular simulation to study the mechanical behaviour of MMMs. The novelty of the approach is using the statistical associating fluid theory (SAFT-γ Mie) model to correlate experimental thermophysical properties of small molecules and produce reliable and versatile CG molecular parameters for large systems such as polymers [1]. These parameters can be directly used for Molecular Dynamics (MD) simulations. To parameterise the CG model of single-walled carbon nanotubes (SWCNTs), we used the Morse and Tersoff potentials. For the polyethylene, we used appropriate experimental data to obtain the intramolecular parameters and classical force fields for the intermolecular parameters through MD simulations. The results show that both CG models are suitable for reproducing the mechanical response of individual polymers and SWCNTs compared to the atomistic results. Additionally, the findings suggest that long SWCNTs may enhance the tensile strength of MMMs compared to a pure polymeric matrix. In contrast, short nanotubes have no significant effect on their mechanical response.

Acknowledgments
The authors gratefully acknowledge the support of the RCGI, Shell Brazil, USP, FAPESP (2020/01558-9 and 2022/06973-0), and Imperial College London.

References
[1] M. Fayaz-Torshizi, E.A. Müller, Coarse-Grained Molecular Simulation of Polymers Supported by the Use of the SAFT-γ Mie Equation of State, Macromol Theory Simul. 31 (2022).

Nanocarbon obtained from lignin pyrolisis as a reinforcing agent in polyamide-6 nanocomposites

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Nanocomposites based on polyamide-6 (PA-6) reinforced with graphene oxide (GO - Graphene Oxide) have been the subject of intense research due to their special characteristics, providing great industrial applicability in several areas, such as the civil construction industry, electronics and automotive sector. Studies focused on these nanocomposites are typically associated with the improvement of their mechanical properties (tensile strength and modulus of elasticity) and thermal properties. The aim of this work is to manufacture an alternative PA-6 nanocomposite material, using lignin as a graphene precursor, after going through temperature/pressure processes aiming to transform the lignin into a "lignin nanocarbon" to act as a reinforcing agent, as well as to determine the optimal formulation of PA-6/GO capable of obtaining the desired reinforcement effect for applications of these nanocomposites in structures to be used by the automotive industry.

POLYANILINE-SILICA DOPED WITH OXALIC ACID AS A NOVEL EXTRACTOR PHASE IN TF-SPME FOR DETERMINATION OF HORMONES IN URINE SAMPLES USING 96-WELL PLATE SYSTEM AND HPLC-FLD DETECTION.

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Polyaniline (PAni) is an intrinsically conducting polymer (ICP) that has attracted much interest in different areas of science, mainly due to its unique characteristics, which include easy preparation, low cost, high stability and processability [1]. These characteristics allow researchers to easily carry out modifications of the structure by changing different synthesis parameters. This particularity gives polyaniline a wide range of technological applications, such as supercapacitor, electromagnetic shielding, gas sensor and biosensor, and it has also been used as the extractor phase for sample preparation techniques [2]. Complex samples, such as urine samples, need a sample preparation step prior to instrumental analysis and the determination of hormones in these samples is important because hormonal concentration changes in women have been associated with breast, endometrial and ovarian cancers, and they are also relevant for prenatal diagnosis [3]. The aim of this study was to evaluate the applicability of different polyanilines for the analysis of three hormones, including 17-β-estradiol (E2), 17-α-ethinylestradiol (EE2) and estrone (E1) in urine samples using the thin film solid phase microextraction (TF-SPME) technique associated with 96-well plate system and detection by high-performance liquid chromatography coupled to fluorescence detector (HPLC-FLD).

Acknowledgments:

The authors are grateful to the Brazilian Government Agency Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) for the financial support which made this research possible.

References:

[1] S. Bhandari, Polyaniline Blends, Composites, and Nanocomposites, 2018.

[2] A. Amiri e C. M. Hussain, Conductive Polymers in Analytical Chemistry, vol. 1405, 2022.

[3] M. Adeel, X. Song, Y. Wang, D. Francis e Y. Yang, Environmental International, 2016.

Production of air filters composed of PHBV/nanoclay fibers for enhanced indoor air quality

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Air filters are devices designed to eliminate harmful particles from the air, enhancing indoor air quality and decreasing respiratory illnesses[1]. Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV)-based air filters can provide an environmentally friendly alternative to traditional filters composed of non-biodegradable materials since PHBV is a natural and non-toxic polyester[1]. Incorporating nanoclays into these filters can enhance their filtration capacity compared to commercial ones due to their high surface area and electrostatic charges[2]. Besides, the use of nanofiber as a potential material for composite-air filter media is a promising alternative[2]. Therefore, this work aimed to produce PHBV/nanoclay non-woven fibrous membranes for air filter applications. The filters were produced through the solution blow spinning (SBS) technique varying the polymer concentration (3-5 %m/v), bentonite clay concentration (0-0.5 %m/v), and spinning pressure (70-140 kPa). The statistical influence of the factors was assessed by analysis of variance (ANOVA). The morphology (SEM), porosity, grammage, pressure drop, and filtration efficiency of the air filters were also evaluated. The results indicated that higher PHBV concentration favored the formation of micrometric (1.7 μm) fibers, whereas spinning pressure decreased the diameter to a minimum of 0.4 μm. There was no direct statistical influence of nanoclay concentration on fibers′ diameter. The PHBV/nanoclay filter presented filtration efficiency for small airborne particles of 98% and a pressure drop of 23 Pa, which were superior properties than those of the commercial filters with higher grammage.

Acknowledgments:

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brasil (CAPES) – Finance Code 001 and CNPq.

References:

[1] A.O.Basar, C.Prieto, M.Pardo-Figuerez and J.M.Lagaron, 8, 3798-3811 (2023)

[2] M.Makaremi, R.T.Silva and P.Pasbakhsh, 119, 7949-7958 (2015)

Recent advance in bone tissue regeneration

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The aim of the current work is to improve the implementation of existing cell / bone substitute therapy for the serious complication of non-union bone fractures and also to provide holistic transition from conventional regenerative medicine to integrative, personalized regenerative medicine. To this purpose 3D scaffolds attractive for bone repair based on biopolymers (sodium alginate, fish gelatin, kappa-carrageenan) and graphene derivatives were fabricated and investigated under the complex condition envisaged by real-life bone repair application, e.g. biomineralization and morphological changes, structure and in vivo models. CD1 mice models were also used to assess de novo osteogenic potential by ectopic implantation in the subcutaneous dorsum pocket of the animals. The results underline the formation of mineralized extracellular matrix while specific bone markers determination stressed the osteogenic phenotype of the cells. These data support that graphene derivatives bioactivity in osteogenesis mechanisms as being self-sufficient to elevate osteoblast differentiation and bone formation in ectopic sites. Conversely, the advent of improved 3D printing technologies has enabled the engineering of bone tissue for patient-specific healing. Our aim was to develop novel printable formulations using our previous experience based on different biolopolymer e.g. gelatin/pectin polymeric matrix that integrates synergistic reinforcement effect graphene oxide and oxidized nanocellulose fibers. Using 3D printing technology and the aforementioned composite inks we fabricate bone-like scaffolds with enhanced compressive modulus, excellent printability, scaffold fidelity and high perspectives for bone tissue engineering applications. The authors acknowledge the financial support from the Ministry of National Education, Executive Unit for the Financing of Higher Education, Research, Development and Innovation (UEFISCDI), project number PCE 103/2022.

Rietveld Refinement, electrical and mechanical study of PVA/ $LiClO_4$ flexible membranes.

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Solid electrolytes have been mainly investigated in the last two decade to be applicable in battery devices. Usually, polymeric matrix blended with ionic salts results in composites, which are used aiming flexibility, stability and electrical performance, beyond being biodegradable materials[1,2]. In this work PVA (polyvinyl alcohol) blended with $LiClO_4$(lithium perchlorate) flexible membranes have been prepared, they were performed based on Solvent Casting Method. The samples were described by DMA and strain-stress measurements. They specify favorable elastic energy storage under mechanical stress. The complex impedance spectrum shows that there is a decrease in the resistance values (order of $10^3 \Omega$), up to 5% of $LiClO_4$, then, a good electrical performance. Rietveld refinement method has been done using the experimental PXRD. Is easy to see that $LiClO_4$doesn't contribute in the diffractogram profiles due to its dissociation in the synthesis samples andthe background intense decrease to 5% of lithium perchlorate, indicating an efficiency of the PVA polymerization. Therefore, we concluded that the material obtained becomes promising to keep being studied and for applications in technological devices.

Acknowledgements:
The authors would like to thank FAPEAM Brazilian agency for the financial support.

References:
[1] R. Putri, C. Sundari et al., Journal of Non-crystalline Solids vol. 556, page 2 (2021).
[2] S. Abarna et al. Materials Science-Poland vol.37, page 331-337(2019).

Self-Healing and Electrical Behavior of Reduced Graphene Oxide-Doped PEI/PAA LbL Films

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Developing new materials for organic electronics is essential for advancing technology in fields such as soft robotics, biomedical devices, and sensors. Self-healing materials with good electrical transport properties are highly desirable for all these applications, which can be achieved by forming nanocomposites. This study explores the self-healing and electrical behavior of a polymer matrix doped with reduced graphene oxide (rGO) nanoplates. The matrix film is obtained by the layer-by-layer (LbL) deposition of poly(ethyleneimine) (PEI), poly(acrylic acid) (PAA), and functionalized rGOs. Impedance magnitude curve is acquired for each layer deposited during the LbL film assembly and I vs V curve after deposition. The high water affinity of the PEI/PAA matrix strongly influences the electrical behavior of the composites, which we investigated by conducting experiments at different humidity levels. The I vs V measurements were acquired in three potential ranges with sweep cycles ranging from -V to +V, for V = 2, 3 and 5V in two distinct voltage steps, indicating hysteresis. We observe that the current increases in each voltage cycle, suggesting the presence of charge carrier traps that could be used to form a memristor. Overall, our findings provide insights into the properties of reduced graphene oxide-doped polymer matrices, with potential applications in the development of self-healing organic electronics.

Acknowledgment:

The authors are grateful to FAPESP, CNPq, and CAPES for financial support. Experimental support from the Microfabrication Laboratory (LMF-21855) at Brazilian Nanotechnology National Laboratory, CNPEM.

References:

Shape memory effect of graphene-based thermoplastic polyurethane nanocomposites: influence of annealing-induced phase separation

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In this study, 0.1 wt.% of graphene nanoplatelets (GNP) or 0.1 wt% of multilayers graphene oxide (mGO) are incorporated into TPU matrix using solution casting processing, and the contribution on the phase separation of these domains is observed. This phenomenon is even more pronounced when graphene-based nanocomposites are submitted to annealing at 110 ºC for 24 hours, suggesting a good interaction between the GO and GNP with the hard and soft segments, respectively. After annealing, the nanocomposites present better performance in shape memory effect regarding the increase on shape recovery ratio (Rrec) of around 9% compared to the non-annealed TPU. All nanocomposites maintained a high strain during SME programming, higher than that of the pure TPU, before and after annealing, indicating a direct influence of the graphene on the shape memory effect.

Acknowledgments

The authors would like to acknowledge CAPES for the grants PROSUC - 88887.154374/2017-00, and PRINT 88887.310339/2018-00. The authors also acknowledge the FAPESP through grants 2012/50259-8, 2017/07244-3, 2018/10910-8, and 2023/02043-0 as well as the MackPesquisa, Project nr. 181009 and the FIPT for the New Talents Program grant. The work was also partially supported by European Commission, within the framework of the H2020-MSCA-RISE-2016-734164 Graphene 3D project. The authors also thank Delmac do Brasil for supplying the polymer.

References

[1] N. Meinzer, Nat. Rev. Mater., 2, 2017, (2017)
[2] F. Pilate, A. Toncheva, P. Dubois and J. Raquez, Eur. Polym. J., 80, 268-294, (2016)
[3] F. C. F. Valim, G. P. Oliveira, G. da Cunha Vasconcelos, L. B. de Paiva, C. Santillo, M. Lavorgna and R. J. E. Andrade, J. Appl. Polym. Sci., 139, 1-13, (2022)

Socially and environmentally sustainable design of composite structures using natural Brazilian fibers

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This work presents two projects using natural fibers and polymer matrix composites that consider the impact of the materials used in the communities and the environment. The first project focuses on the design and manufacturing of an urban bicycle frame with a recyclable PET core material reinforced with banana fiber and castor oil polymer matrix. The second project explores the design of a prosthesis for para-athletes with a sisal fiber reinforcement and epoxy matrix. The paper discusses the rationale behind the selection of the design using geometric and parametric optimizations, the properties of the fiber and composite laminate found in the literature, the manufacturing process using Resin Transfer Molding (RTM), and the feasibility and scalability of the product. A prototype of the bicycle and the prosthesis were built to compare the fully handmade process with an industrial process with greater resources. The paper shows experimental test data and comparison with the simulated performance from the design phase. The results show that despite the challenge in manufacturing composite structures with such unconventional materials, the solutions presented structural integrity within a predictable range. These results are a significant step towards the use of natural fiber composites as a socially and environmentally sustainable alternative to more classical synthetic composites.

The impact of carbon nanoparticle dispersion and concentration on IPN-based nanocomposites' properties

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Conductive polymer coatings are in high demand due to their versatile properties. They are especially interesting for the airspace industry, which could employ them as lighter electromagnetic shielding and lightning strike protection devices for modern aircraft frames 1. Graphene and related nanomaterials, such as CNTs, have been extensively employed in the fabrication of conductive polymer materials. Adding them to a homogeneous polymer matrix is the most common approach, but using immiscible polymer blends can lead to much lower percolation thresholds due to the double percolation phenomenon: by selectively dispersing the nanofillers into a minor continuous phase or placing them at the interface between two co-continuous phases, their concentration can be locally increased 3. Within this framework, interpenetrating polymer networks (IPNs) are a class of polymeric systems composed of two or more immiscible polymers with no covalent bonds between their chains and that are interlocked on a molecular scale. Poly(ether imide) (PEI) is typically used in combination with epoxy resins to form IPNs, resulting in a biphasic morphology split between epoxy- and PEI-rich phases 3. This morphology not only leads to better mechanical and thermal properties but also allows for double percolation phenomenon to take place. The addition of GNPs and CNTs in IPN systems have not been widely studied in literature,and the dispersion aspect of the sample fabrication has been largely ignored. By comparing ultrasonic- and mechanical-based dispersion routes, we have established the best approach for fabricating carbon nanoparticle-reinforced epoxy/PEI IPN nanocomposites and investigated their electrical and mechanical properties.

References:
1. Gagné, M. Therriault, D. Prog Aerosp Sci 64, 1-16 (2014)
2. Ma, C. G., Xi, D. Y. Liu, M. J Compos Mater 47, 1153-1160 (2013)
3. Farooq, U., Teuwen, J. Dransfeld, C. Polymers (Basel) 12, (2020)

The Role of Carbon Nanotubes Dispersion in PVDF/CNT Electrospun Nanofibers

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Conductive electrospun fibers are made using conductive nanofillers, such as carbon nanotubes (CNTs), carbon black or graphene mixed with a spinnable polymer [1,2]. However, the nanofiller agglomerates interfere on the spinnability, consequently the fiber quality and its electrical conductivity. This is a challenge in order to produce suitable flexible electronic devices. Thus, in this project we studied the incorporation of carbon nanotubes (CNTs) in polyvinylidene fluoride (PVDF) nanofibers using electrospinning in order to form an electrically conductive structure [3]. PVDF and CNT were dissolved and dispersed to obtain an electrospinnable solution of dimethylformamide (DMF) and acetone. This mixture was then electrospun using a drum collector in order to produce the aligned nanofibers. The CNTs percolate to form a conductive network resulting in electrically conductive nanofibers. The percolation threshold was found to be time-dependent. The time-dependency of the percolation threshold shows that a lower CNT concentration can be used to achieve electrical percolation while avoiding agglomeration, thereby helping to manufacture electric devices that require conductive nanofibers. As the percolation depends on both concentration and dispersion time, the rheological properties must be well understood in order to achieve the desired material properties.

References:

[1] J-M.Park, et al., Advanced Composite Materials 25:4, 305-316 (2016)
[2] S.Mirjalali ,et al., Macromol. Mater. Eng. 308, 2200442 (2022)
[3] S.Sharafkhani and M.Kokabi, Composites Part B: Engineering 222, 109060 (2021)

4D Printing of Thermoresponsive Hydrogels

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Printable 4D materials dynamically change their shape in response to external stimuli, such as temperature, pH, or light. As a result, it has the potential to enable the development of smart materials and self-assembly systems for various applications [1]. Poly(N-isopropylacrylamide) (PNIPAm) is a promising hydrogel for 4D printing, as it undergoes reversible volumetric chances in response to temperature. This thermoresponsive behavior allows the material to adapt to environmental fluctuations and can be used to create structures with controlled geometries. Alas, the poor mechanical strength of PNIPAm can hinder its application for printing. To bypass this problem, one can develop double network systems, although these modifications could potentially impact the thermal-responsivity of PNIPAm. The addition of inorganic compounds, such as boron nitride nanostructures, could minimize the impact on the thermoresponsiveness. Boron nitride nanosheets (BNNS) are 2D materials that can be incorporated in hydrogels to improve its overall properties [2]. It exhibits biocompatibility, excellent mechanical strength, chemical stability, and high thermal conductivity. As the thermal response is related to the polymers' thermal conductivity, it is expected that the BNNS could modify this property. Thus, this work evaluates the influence of the BNNS on the thermal responsiveness of the printable nanocomposites. Functionalized BNNS were incorporated into fixed content hydrogels at different concentrations, and their distribution within the matrix was analyzed using SEM. FTIR was used to evaluate the chemical structure, and DSC was employed to visualize thermal events.

Acknowledgements:
Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES).References:

[1] Champeau, M., Heinze, D.A., Viana, T.N., de Souza, E.R., Chinellato, A.C., Titotto, S., 2020. Adv Funct Mater 30, 1910606.

[2] Lima, D.M., Chinellato, A.C., Champeau, M., 2021. Soft Matter.

Anionic block amphiphilic copolymer synthesis by RAFT for the generation of antimicrobial particles

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The synthesis of block polymers the combination of different repetitive units for the planning and control of characteristics such as molecular weight and chain architecture, enabling new studies and the creation of unprecedented polymeric materials for a variety of applications [1]. In this work, we report the syntheses by RAFT of amphiphilic block copolymers the oriented towards the generation of antimicrobial systems. All syntheses were monitored by GPC and characterized by FTIR. The aggregation of the final amphiphilic materials in aqueous media was analyzed by DLS. The syntheses were performed in two steps. Firstly, the macroCTA of PS was obtained followed by the second block of PtBA. The PS-b-PtBA copolymers were hydrolyzed to provide the amphiphilic PS-b-PAA. GPC monitoring indicated that reactions have occurred in a controlled manner. Characterization by FTIR confirmed the chemical structure of the materials. In pH 7 aqueous solution, copolymers aggregated into particles presenting average hydrodynamic diameter of hundreds of nanometers 345 ± 31 nm by DLS. It has been shown that the method based on RAFT was able to generate designed amphiphilic block copolymers with systematic changes on the molar mass and composition. The PS-b-PAA copolymers, with different ratios between the blocks content, will be subsequently tested for their antimicrobial action, whether or not bound to copper cations or others.

Acknowledgements:

CAPES and FAPESP

References:

KANG, S., KIM, G. H., and PARK, S. J. Conjugated block copolymers for functional nanostructures. Accounts of Chemical Research, v. 55, n. 16, p. 2224-2234, 2022.

Anticrusting membranes of polyurethane with zirconia nanoparticles

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Industrial oily wastewater and oil spills have a negative impact on the environment and human health, attracting attention in development of new technologies. In this study, polyurethane (PU) membranes were produced using the phase inversion technique, for pure polymer and additive with zirconia nanoparticles (NPs) incorporated into the polymeric matrix. The surface of the NPs were functionalized with oleic acid. The incorporated NPs were characterized in terms of crystallinity, particle size, and surface functionalization. The membranes were structure characterized by microscopy and the permeability using aqueous fluid. The crystalline and particle size characterizations revealed that the NPs have a monoclinic crystal phase and an average particle size of 6 nm. Microscopy images revealed that the incorporation of NPs in membranes results in porosity controlled by additive. Permeability tests indicated that the incorporation of NPs reduced membrane permeability, but with a steady flow, indicating that the NPs content change membranes structure, preventing compaction compared membrane without NPs additive. The results suggest that membrane porosity can be altered through phase inversion, with NPs incorporation being key to obtaining resistant and efficient separation membranes. Therefore, this study demonstrates the potential of adapting the PU matrix with oxide NPs membranes for use in emulsion separation processes.

Acknowledgments:CAPES, FAPES, CNPq e FINEP-ANP PRH 53. 1

References

Vatanpour, V. *et al. (2022), Science of The Total Environment*, 810, 152228. https://doi.org/10.1016/j.scitotenv.2021.152228.

Gu, H. et al. (2020), Applied Surface Science, 512, 144837. https://doi.org/10.1016/j.apsusc.2019.144837.

Barrier properties of starch films with the addition of different concentrations of buriti oil (Mauritia flexuosaL.)

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Films obtained from renewable and biodegradable biopolymers, such as cassava starch, have received great attention related to their environmental impacts and use in the food packaging industry. In this study, active biodegradable cassava starch films with antioxidant capacity through the incorporation of buriti oil were produced. The films were developed using the casting technique (4 g of starch/100 mL of water) with the addition of glycerol asa plasticizing agent (0.25 g of glycerol/g of starch), the active compound was incorporatedat concentrations of 5% and 10% [1]. The films were characterized for barrier properties (moisture content, water solubility, water vapor permeability and light transmission) [2]. The results confirmed that the addition of buriti oil modified the properties, obtaining films with greater color intensity, better barrier to light transmission, and reduced permeability to water vapor. This behavior is associated with the hydrophobic nature of the active compound incorporated into the polymeric matrix, as well as the presence of natural antioxidants in the oil, such as carotenoids. A reduction in the moisture content and water solubility was also observed, which favors a greater application of these materials as packaging for different food matrices.

Acknowledgements:The authors thank to Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) (407371/2021-3) and Fundação de Amparo à Pesquisa do Estado deMato Grosso/Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (FAPEMAT/CAPES) (473/2022). References:[1] Assis et al., Industrial Crops and Products, 109, Pages 818-827, 2017[2] ASTM E96-95, Standard Test Methods for Water Vapor Transmission of Materials, 2000

Biodegradable packaging of cellulose acetate incorporated with pequi oil (Caryocar brasilienseCamb.)

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The development of films from biopolymers has been studied to replace or reduce the use of synthetic polymers and conventional plastic packaging, associated with the interest in increasing food stability from the addition of bioactive compounds to the polymeric matrix. In this context, cellulose acetate was used to produce active films with different concentrations of pequi oil (5% and 10%). The films were obtained from the technique and the effect of oil incorporation on barrier properties was evaluated [1,2]. According to the concentration of added oil, there was a gradual increase in the color intensity of the films, associated with the presence of carotenoids (such as β-carotene), with a significant reduction in UV-Vis light transmission. There was an increase in the opacity of the films, of 0.33 ± 0.03 Abs600nm/mm, 2.18 ± 0.02 Abs600nm /mm, and 2.85 ± 0.05 Abs600nm /mm, for the control film and films with 5% or 10% oil, respectively. As expected, the hydrophobic character of the oil led to a reduction in water vapor permeability, from 0.32 ± 0.02 g mm m-2h-1 kPa-1 to 0.12 ± 0.02 g mm m-2h-1 kPa-1, for the control film and the film withthe highest concentration of antioxidant additive. Pequi oil can be an additive used to obtain biodegradable packaging with antioxidant capacity and a better barrier to the light transmission, which demonstrates the potential stability of foods susceptible to photodegradation.

Acknowledgements:The authors thank to Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) (407371/2021-3) and Fundação de Amparo à Pesquisa do Estado deMato Grosso/Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (FAPEMAT/CAPES) (473/2022). References:[1] Assis et al., Industrial Crops and Products, 147, page 112212, 2020[2] ASTM E96-95, Standard Test Methods for Water Vapor Transmission of Materials, 2000

Biopolymeric films loaded with hybrid nanoplatforms based on Carbon Polymer Dots and Halloysite

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Carbon polymer dots (CPD) are a novel type of fluorescent, carbon-based nanomaterial (0D) with a of less than 20 nm. Due to their unique properties, CPD have potential applications in various fields of interest, such as optical, electronics, sensors, or biomedicine [1]. On the other hand, another promising material is clay minerals, which can be combined with diverse species and form composites with low toxicity, increased biocompatibility, and improved mechanical stability [2]. Within this perspective, clay minerals can also be combined with polymers which can be processed as functional composite films. In this study, alginate (A) and carboxymethylcellulose (CMC) polysaccharides blend was combined with a hybrid material developed by the union of Halloysite clay mineral (HC) and CPD at different concentrations to form reinforced biodegradable films. The films were characterized by diverse physicochemical techniques, and their barrier and mechanical properties were investigated. The FTIR spectra of the films indicated strong hydrogen bonds between the components of the material, at the same time that the introduction of hybrids into the biopolymeric matrix significantly blocked a substantial portion of the UV radiation due to the ability of the CPD to absorb ultraviolet radiation and blue photons [3]. In addition to good mechanical properties, the water vapor transmission rate of the resulting films showed a decrease of almost 2 times in the composite films compared to the pure polymer film, indicating that the addition of HC-CPD hybrid improved the barrier properties against water vapor.

Acknowledgements:
FAPEMA, UFMA.

References:

[1] C. Xia, S. Zhu, T. Feng, M. Yang, B. Yang, Advanced Science, 6, 1901316 (2019)
[2] J. S. Boruah and D. Chowdhurry. Minerals, 13, 26 (2022)
[3] Y. Han, Y. Wang, B. Zhao, Y. Bai, S. Han, Y. Zhang and W. Yu, Advanced Composites and Hybrid Materials, 6, 39 (2023)

Carboximethylchitosan/*Jatropha mollissima* compoundfor hemostatic application

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Traumatic injury is responsible for more than 5.8 million deaths/year, although there are considerable advances in the area of hemostatics, blood loss is one of the biggest causes of death in hospitals. The development of optimized hemostatic agents can be an alternative to minimize this problem, as is the case of the biomaterial based on O - carboxymethyl chitosan with the addition of Jatropha mollissima, objective of this work. The o-carboxymethylchitosan was synthed from chitosan and later, the nanoparticles were obtained by the ionic crosslinking method. The ethanolic extract of the sap was obtained by the ionotropic gelation technique. The O - carboxymethyl chitosan solution was obtained ata concentration (0.25 m/v), added with 1.25% (m/v) of the ethanolic extract of jatropha mollissima sap. The raw material was characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Solubility. The nanoparticles were characterized by Dynamic Light Scattering (DLS); Zeta potential (PZ), UV-vis and hemostatic activity. In the FTIR, it was possible to observe the formation of characteristic groups present in O - carboxymethyl chitosan, in the ethanol extract of the sap, the presence of groups related to flavonoids, terpenes and amino acids was observed. In the evaluation of the solubility, both of the carboxymethylchitosan and the extract, it was observed that both are soluble in water. In the DLS, it was found that the nanoparticles present homogeneity and an average of 265.24 ± 2.30. In PZ, the nanoparticles suspension showed an average instability of -16.77 nm, which is desirable for application as hemostatic for stabilizing bleeding. In UV-vis, the encapsulation rate had an efficiency of 12.5%, which did not interfere with the hemostatic effectiveness in vivo. Based on the results, it can be concluded that it is possible to obtain nanoparticles of O - carboxymethylchitosan/jatropha mollissima with potential for use as a hemostatic agent.

CHARACTERIZATION OF A NEW POZZOLANIC BINDING INGREDIENT ORIGINATED FROM THE MIXTURE OF CLAY AND SUGAR CANE LEAF.

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Portland cement is one of the most widely used materials in the construction industry, but its production is highly associated with greenhouse gas emissions, raising environmental concerns and pressures for sustainable alternatives. In this context, the use of agro-industrial waste such as sugarcane leaves has been proposed as a viable solution for the production of alternative binders. This study presents a new pozzolanic binder obtained by combining sugarcane leaves and clay (CFCA), in which the former is used as fuel in the calcination process, integrating these two pozzolanic sources and providing satisfactory benefits. CFCA was subjected to grinding and sieving processes. Mortars were produced with partial substitutions of Portland cement by CFCA in different proportions (control, 10%, 20%, 30%, 40%, and 50% by weight), evaluating the consistency, which reduces as the substitution increases. The mechanical strength of the mortars before and after exposure to fire at a temperature of 600°C for two hours resulted in a decrease in compressive strengthin the increasing order of Portland cement substitution by CFCA, being from 35% to 43%. However, even with this reduction, the results still indicated that it is possible to substitute Portland cement with CFCA up to 50% by weight, resulting in approximately 23 MPa after exposure to fire, showing that the use of CFCA can be a promising and sustainable alternative in the production of binders for the construction industry.

Acknowledgement:
I would like to acknowledge the Coordination for the Improvement of Higher Education Personnel (CAPES) in Brazil for their support in making this work possible.

References:
[1] A, Mahmoud; S, Farhan. Sustainable cement and concrete production using partial replacement of industrial waste materials. J. clean prod., v. 237, p. 117717, 2019.
[2] N, Abdelghani; H, Meftah. Influence of metakaolin on the properties of geopolymer pastes and mortars. Constr Build Mater., v.47, p.1211-1218,2013.

Characterization of rotary-jet spun Polycaprolactone/Chondroitin Sulfate (CS) blends with incorporated lyophilize Bone Powder

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The developing of bioactive scaffolds to bone repair has been an alternative, but suffers from limitations regarding the extent of bone loss and the morbidity of the donor site [1]. Poly(ε-caprolactone) (PCL) has good biocompatibility and biodegradability, suitable for making bone scaffolds. However, its smooth surface and strong hydrophobicity generate low biological activity, not being viable for adhesion of osteoblasts and regeneration of bone tissue when used alone, requiring the addition of materials that corroborate the desired cellular viability.[2][3]. In the present work, rotary jet spun PCL/Chondroitin Sulfate containing different concentrations of lyophilized bone powder were produced for biomedical applications. So far, morphological and chemical characteristics have been evaluated. For the next steps, mechanical tests, in vitro and in vivo, will be performed, in addition to other characterizations to check the properties of each group. After that, all other results will be analyzed, in order to verify the functionality of the material and applicability in Bone Tissue Engineering.

Acknowledgements:
Thanks for Interdisciplinary Laboratory of Advanced Materials (LIMAV).

References:

[1] Machado-Paula M. M. et All. Rotary Jet-Spun Polycaprolactone/Hydroxyapatite and Carbon Nanotube Scaffolds Seeded with BMMS Cells Increase Bone Neoformation ACS Applied Bio Materials 2022 5 (3), 1013-24 DOI: 10.1021/acsabm.1c00365

[2] Kong, X. et al. Chondroitin Sulfate/Polycaprolactone/Gelatin Electrospun Nanofibers with Antithrombogenicity and Enhanced Endothelial Cell Affinity as a Potential Scaffold for B V T Engineering. Nanoscale Res Lett 16, 62 (2021). https://doi.org/10.1186/s11671

[3] Andrade, T. M. et al. In vitro and in vivo evaluation of rotary jet spun poly (ε-caprolactone) with high loading of nano-hydroxyapatite. Journal of Materials Science: Materials in Medicine, v. 30, n. 2, p. 1-14, 2019.

Characterization strategies to investigate ionizing radiation effect on nanocarbon material

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Advanced carbon-based materials, such as carbon fibers and other nanostructured forms of sp^2carbon, will enable revolutionary applications, as long as one can promote good dispersion in substrates and matrices. Electron beam irradiation can induce structural modifications leading to point of defects, voids and/or displacements and establish active sites to attach desired functional groups to improve this dispersion. The energy threshold for incident and tangential electrons capable of creating these changes is 15 eV to 22 eV and 33 eV to 44 eV, respectively [1]. Despite this complexity, this work will present results of characterization techniques that can help to evaluate the effect of ionizing radiation on carbon nanofiber structure. TGA, XRD and XPS techniques were used to assess the modification on carbon nanofiber (CNF) structure when submitted to direct radiation grafting process. TGA results showed that this process in solution of acrylonitrile (AN) 40% v/v obtained a high yield of nitrogen grafted on CNF surface, which is in accordance with XPS results. Direct radiation grafting of CNF in solution of 40, 60 and 80% v/v of AN presented nitrogen content of 13.5%, 10.2% and 7.2%, respectively. There is also clear indication that the sp^2-type carbon is being disrupted, showed by the reduction of sp^2/sp^3rate from 8.93 to 0.76 for CNF immersed in AN solution not irradiated compared to the irradiated one at same conditions.

Acknowledgments: We would like to thanks Kulun Hong of Oak Ridge National Laboratory for the XPS results and Dr. Roberto Uribe of Kent State University for the radiation experiments.

Reference:
[1] T. Filleter and H.D. Espinosa, Carbon 56, 1-11 (2013)

Comparative study of the behavior pattern between a self-compacting concrete with the use of rice husk ash and a vibrated concrete

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Recently, several works have been carried out with the use of rice hull ash (RHA)in the production of concrete, and a lot of self-compact concrete (SCC) has been produced. However, with this objective, concretes were produced with the use of RHA, but this material is industrialized, in the content of 35% in relation to the cement in addition, was varied and the relationship "water/cement" ; in relation to the "superplasticizer/cement" with was constant. Two series of vibrated concrete were also produced, for comparative purposes, one varying the "water/cement" ratio by 0,4; 0,5 and 0,6 and keeping the relationship "coarse aggregate/total mass of concrete" and another one keeping the relationship "water/cement" at 0,5 and varying the relationship "coarse aggregate/totalmass of concrete" . Note that the RHA was suitable for manufacturing the SCC, so that theymet the requirements in the fresh state, and in the weathered state. Validation tests werecarried out for the SCCs in the fresh state, Slump-Flow, V-Funnel, and the Case L [1], segregation tests by sieving, and the evaluation of the apparent dry specific mass. Both forcases of SCC and for cases of vibrated concrete, tests were carried out in the state of resistance, simple resistance, diametral resistance, modulus of elasticity, water absorption tests at 28 days of curing. The SCC mechanical strength values remained higher, despite thehigher values for the "water/cement" relationship, and the mechanical strength values relative to the absorption values remained more consistent for SCC. Also-note that self- compacting concretes showed signs of greater ductility than vibrated concretes.

REFERENCES:

[1] Bispo R. A., Vicente G. O., Júnior G. P. S., Benjamim D. U., M. A. M. Alcântara. Investigation of rheological behavior of self-compacting and high-performance composite concretes. Materials Research. 24 (suppl. 2); 2021.

Comparative study of the mechanical properties of Portland cement mortar and cementicious composites reinforced with cellulose in civil construction

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In civil construction, several types of materials are used in the works, such as cement. However, it has factors that compromise the application and lead to undesirable pathologies in constructions. Therefore, to improve the performance of these materials, several studies aim to improve Portland cement with the addition of cellulose microfibers, which is a type of renewable and sustainable resource. With this, it can improve the quality of mechanical properties and durability, making it possible to make buildings more efficient. In this way, this study aims to carry out a previous research of the mechanical properties of Portland cement mortars and cementitious composites reinforced with cellulose in civil construction, so that the benefits can be seen and analyzed and where this new technique can be improved to assist in the construction. As a Narrative Literature Review study, this work brought an exploratory study, based on articles published from 2003 to 2022. Use of descriptors to delimit the research fields and mortar: Portland cement, composite, cellulose, mechanical property. The following databases were used: Scopus, Web of Science; Scielo, ScienceDirect and Google Scholar. It can be concluded after analyzing all the articles chosen that the use of cellulose in mortars has positive effects on the mechanical and microstructural properties of the materials when used in adequate amounts, as in addition to being a biodegradable and ecologically correct material, it improves resistance to compression, has a higher modulus of elasticity, greater tenacity and durability and resistance to mortar cracking. However, there are still challenges to be overcome.

Compositional evaluation associated with the electrical and electrochemical performance on Ni-modified silicon oxycarbide (SiOC/Ni) ceramics

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Silicon oxycarbide (SiOC) matrices are structurally modified through incorporation of transition metals to polymer precursors, improved formation of electroactive phases (graphitic domains and SiC) when electrical applications are required[1]. This study evaluated different amounts of nickel chloride at the Ni-containing SiOC materials, aiming electrical and electrochemical investigations. Six polymers were synthed by reaction between 60 wt.% poly(methylhydrosiloxane) and 40 wt.% divinylbenzene, containing 20 wt.% polyethylene and addition of 0,1,3,5,10 and 20 wt.% of $NiCl_2$. Ceramics were obtained at 1400 °C under static argon atmosphere with heating/cooling rate of 5 °C/min. FT-IR spectra for SiOC/Ni ceramics revealed three typical bands attributed to νSi-O-Si, νSi-C and δSi-O, confirming the mineralization process. The larger amount of nickel allowed the replacement of oxygen by carbon atoms. Static argon atmosphere combined with greater amounts of $NiCl_2$resulted in a proportional increase of graphitized ceramic. The proportionof β-SiC phase increased up to 5 wt.% Ni and decreased for ceramics with 10 and 20 wt.%. The length of carbon crystallites in-plane increased up to the SiOC 5% nickel and reductionsfor those with 10% and 20%. The resistivity on the powdered ceramics showed promisingalternative as conductive material. The highest and lowest resistivity values were 129.88and 0.44 Ωcm, corresponding to Ni-free SiOC and 5 wt.% Ni SiOC, respectively, so 10 and20 wt.% Ni SiOC showed resistivity of 1.38 Ωcm. Resistivity values depend on thecombination of β-SiC and graphite phases not exclusively on a single conductive phase, arising from different proportions of nickel in a static atmosphere. Furthermore, SiOC/Ni presented larger electroactive area values when compared to glassy carbon electrode,whichbecame promising for development of electrochemical devices.

Acknowledgment
CNPq, UEL

References

[1]MAZO,M.A et al. J.Alloys Compd.2021.

Conductive Blends of incorporation PAni-DBSA in the PVA matrix

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Polyvinyl alcohol (PVA) is a water-soluble synthetic polymer, has interesting mechanical and thermal properties, in addition to being biodegradable and non-toxic1, while polyaniline (PAni) is a conductive polymer sensitive to external stimuli such as light, humidity and pH . The addition of PAni to PVA can increase its electrical conductivity and thermal stability, maintaining its solubility in water, making it useful in the production of flexible electronic materials. The modification of PAni with dodecylbenzenesulfonic acid (DBSA) is a common process that aims to improve the solubility, stability and mechanical properties of the conductive polymer2. The copolymerization of PVA with DBSA-modified PAni can be accomplished in a number of ways, including mixing solutions of the two polymers followed by solvent evaporation. The double-distilled aniline was quickly added to a solution of (NH4)S2O8 solubilized in a 1.00 mol.L-1 HCl solution, the proportion of oxidizing agent:monomer used was 4:1 (n:n). The solution was stirred for 3 hours in an ice bath. The emeraldine salt obtained was placed under strong agitation in a solution of NH4OH 0.1 mol.L-1 for 24 hours, after which the base was filtered under vacuum, washed with distilled water and dried in an oven at 60°C for 48 hours. The heavy base was solubilized in DBSA ata 70:30 (m/m) dopant:polymer ratio. The mixture was left stirring for 48 hours2. To obtainthe composites, PAni-DBSA in proportions 1, 3 and 5% were added to a 10% PVA solution,under stirring for 4h. The presence of functional groups and identification of the chemicalbonds formed were observed using Infrared absorption spectroscopy. The morphological structures of the films were observed using scanning electron microscopy (SEM), the thermal analyzes were analyzed using thermogravimetric analysis (TGA). The electrical properties were characterized by means of electrical conductivity measurements.

Conformational analysis of ionic liquids through residual dipolar couplings probed through polymeric gels as an alignment medium

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NMR in weakly aligned media allows the precise extraction of anisotropic NMR parameters that can then be applied to configurational, conformational or even constitutional structural problems. Among the many alignment media available today, reversibly compressible polymer gels such as chemically cross-linked PMMA[1] or polyacrylonitrile[2] allow particularly easy measurement of not only residual dipole couplings (RDCs) but also residual chemical shift anisotropies (RCSAs). Here we present new crosslinked copolymeric gels that can be swellable in common ionic liquids such as BMIM·NTf2- or BMIM·BF4-. The new developed gels allow the extraction of 1DCH RDCs in the butylmethylimidazolium cation and the determination of their conformational space in the pure liquid. The results were in line with previous reports based on Raman spectroscopy.[3] Additionally, the analysis of the 1DCH couplings in the planar imidazolium ring allowed the assignment of the H4/H5 positions of the ring.

Acknowledgements:

UFPE; the Department of Fundamental Chemistry (DQF); the Coordination for the Improvement of Higher Education Personnel (CAPES) for promoting the scholarship

References:

[1] GAYATHRI, C.; TSAREVSKY, N. V.; GIL, R. R. Residual dipolar couplings (RDCs) analysis of small molecules made easy: Fast and tuneable alignment by reversible compression/relaxation of reusable PMMA gels. Chemistry - A European Journal, v. 16, n. 12, p. 3622-3626, 2010.

[2] CARVALHO, D. S. et al. Chemically cross-linked polyacrylonitrile. A DMSO compatible NMR alignment medium for measurement of residual dipolar couplings and residual chemical shift anisotropies. Journal of Magnetic Resonance, v. 302, p. 21-27, 2019.

[3] HOLOMB, R. et al. Ionic liquid structure: the conformational isomerism in 1-butyl-3-methyl-imidazolium tetrafluoroborate ([bmim][BF 4]). Journal of Raman Spectroscopy, v. 39, n. 7, p. 793-805, jul. 2008.

Development of Biodegradable Active Packaging Containing Chia Seed (Salvia Hispanica L.) Extract Nanoparticles with Antioxidant Function

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From data analysis about food conservation, waste, illness from consuming globally contaminated food [1], and incorrect packaging disposal, the need for the development of biodegradable food packaging that increases the shelf life of food and promotes quality assurance is observed [2]. Therefore, the development of active packaging not only protects the food against mechanical factors but also promotes quality and protection against common chemical factors, such as lipid oxidation in food. The use of biodegradable polymers has been studied in recent years, as it is an area with great potential for development not only in packaging but also in various other sectors. The food packaging industry has been using additives, usually synthetic ones, which not only encase the food but also promote an increase in its shelf life, thus prolonging the time it can be stored and reducing food waste [3]. Therefore, this study aims to develop an active packaging with a biodegradable polymer (polycaprolactone - PCL) containing chia seed extract nanoparticles, due to their high antioxidant potential in their composition, with the function of preventing food oxidation. This film was produced with PCL and the nanoparticles were produced through the nanoprecipitation technique. The formed film was characterized regarding the of the nanoparticles, stability in 30 and 60 days, as well as its antioxidant function, obtaining satisfactory results for its initial application.

Acknowledgments: CAPES - Funding Institution.

References

[1] United Nations (UN), 2020. Every year, 420,000 people die from consuming contaminated food.

[2] BUCCI, D. Z.; TAVARES, L. B. B.; SELL, I. Packaging for the storage of food products. Polymer Testing, v.24, n. 5, p. 564-571, 2005.

[3] SIRIPATRAWAN U.; NOIPHA, S.; Active film from chitosan incorporating green tea extract for shelf life extension of pork sausages, Food Hydrocolloids, v., 27, n. 1, pag, 102 - 108, 2012.

DEVELOPMENT OF POLYURETHANE FOAMS WITH GUARANÁ FIBER WITH ANTIMICROBIAN ACTIVITY

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Polyurethane is a versatile material widely used in various industries, such as construction, automotive, furniture, and footwear. It is characterized by its strength, durability, flexibility, and lightness, as well as being an excellent thermal and acoustic insulator, especially in the form of polyurethane foams (PF). However, PF disposal presents challenges, as they take about 30 years to start degrading and release carbon monoxide when burned. This study aims to explore the effect of substituting polyethylene glycol (PEG), the main polyol in PF composition, with natural fibers from guarana bagasse, and to investigate the incorporation of antimicrobial agents in the foams, comparing two substances with distinct antimicrobial properties: silver nanoparticles (AgNPs) and nano-chitosan (NC). Two distinct methodologies were applied to determine the best combination of fiber and polymeric matrix, differing in the order of reagent addition in the reaction. In synthesis 1 (S1), the fiber was homogenized before adding the other reagent in synthesis 2 (S2), the fiber was added along with the last reagent. For the foam synthesis, guarana bagasse fibers were used, including in natura fibers (NF) and treated fibers (FT) with sodium hydroxide. The substitution of PEG by guarana fibers varied from 5% up to a maximum limit of 30% for PF formation. Foams obtained by methodology S1 exhibited greater homogeneity in their structure compared to those produced by S2. When analyzing the ash content in different parts of the S2 foams, variations in values were observed, suggesting a non-uniform distribution of guarana fibers in the polymeric matrix. Mechanical tests showed promising results, indicating that the partial substitution of PEG by natural fibers from guarana bagasse in PF synthesis and the addition of antimicrobial agents, such as AgNPs and NC, are viable solutions.

Diffusion of chlorides in mortars composed withbamboo leaf ash

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Durability of structures is a constant concern in civil and materials engineering, especially when they are exposed to aggressive environments. In buildings with a concrete structure, durability is profoundly reduced when harmful agents penetrate through the pores of the cementitious composite and alter the microstructure and properties of the materials. The attack by chloride ions leads to severe deterioration of reinforced concrete structures, being cited as the most common mechanism of degradation of these structures [1]. The search for new pozzolanic materials is emerging in view of the need for more sustainable binders and, for this purpose, bamboo leaf ash has been identified as an excellent partial substitute for cement. In the development of new materials, accelerated tests represent a good alternative to reduce time and expenses in material evaluation. In order to evaluate the migration resistance of chlorides in composites mortars with 0, 20, 25 and 30% BLA replacing cement were produced and the accelerated method recommended by NT BUILD 492 was carried out. The migration coefficients in the respective mortars were 11.8, 4.53, 4.72 and 6.07. Mortars with CFB showed much higher resistance to chloride penetration than the reference mortar, especially the mortar with 20% replacement. Other durability tests must be conducted, however, this result indicates that the BLA is a is very promising pozzolanic material that allows to obtain cement based materials more sustainable materials decreasing CO2 footprint and with very good durable behavior against chloride penetration.

References

[1] A. Neville, Chloride attack of reinforced concrete: an overview, Mater. Struct. 28 (1995) 63-70. https://doi.org/10.1007/BF02473172.

Effect of drying on the encapsulation of the entomopathogenic fungus *Beauveria bassiana* in polymeric matrix

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In agriculture, nanosystems used as bioinsecticides, such as polymeric nanocomposites, have been successfully used in insect-pest management. When promoting the encapsulationof biological agents, such as entomopathogenic fungi, the objective is to preserve the inherent characteristics of the active agent. Nanocomposites containing alginate(biopolymer) and different concentrations of bentonite (lamellar silicate) exert a barrier effect responsible for modifying the release of the fungus, giving it protection [1]. Thus, beads-type encapsulation matrices of the entomopathogenic fungus *Beauveria bassiana* composed of alginate (1%) and bentonite (2, 4 and 6%), were obtained by ionotropic gelation and dried by air flow or freeze-drying, through fast and slow freezing.The formulations were characterized by: XRPD and FTIR, average particle size, intumescence index and in vitro release. The increase in the concentration of lamellar silicate conferred improvements to the mechanical properties of the beads, ensuring the preservation of the bioactive in formulations submitted to freeze-drying. With this, the preparations with concentrations of 4 and 6% of bentonite and dried by airflow stood out,showing promise in the development of a solid formulation of the bioinsecticide.

Acknowledgments:
Federal University of Alagoas, Fapeal, CNPq, Capes.

References: [1] BATISTA, D. P. C. et al. Encapsulation and release of *Beauveria bassiana* from alginate-bentonite nanocomposite. Royal Society of Chemistry, 7, 26468, 2017.

Electrical, structural and first-principles calculations (DFT) properties of flexible membranes of PVA/ LiOH electrolytes for battery applications.

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Since the advent of solid-state batteries many materials have been used as electrolytes to get better ionic conduction through its crystalline structure. Some types of polymers are interesting to be used in battery components because they combine high mobility of charge carriers and flexibility [1]. For this purpose, in recent years, composites with lithium salt have been use achieving a significance increase in the electrical properties [2,3]. In this work, flexible composites have been prepared, PVA (polyvinyl alcohol) blended with LiOH (lithium hydroxide monohydrate) in the following proportions, 0, 5, 7 and 9% in weight. It was prepared via Solvent Casting Method. As results, X-ray diffraction patterns shows the reflections of monoclinic unit cells of PVA and the increase of the crystallinity due the ion Lithium adsorption, while DFT study reveals changes of charge and atoms distances between $Li^+$and oxygen. The complex impedance spectrum indicated that there is a decrease in the resistance values of the composites up to 7% of LiOH reaching a solubilitylimit. Therefore, we concluded that the material obtained becomes promising for applications in technological devices.

Acknowledgements:
The authors would like to thank FAPEAM Brazilian agency for the financial support.

References:
[1] R. Putri, C. Sundari et al., Journal of Non-crystalline Solids vol. 556, page 2 (2021).
[2] M. Hema et al. Journal of Non-Crystalline Solids vol.355, page 84-90 (2009).
[3] M. Pandey et al. Journal of Polymeric Materials and Polymeric Biomaterials vol. 65, page 759-768 (2016).

Electropolymerization of Conjugated Polymers Applied for Latent Fingerprint Development

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Conjugated polymers are a class of materials that have attracted considerable attention in terms of research and development of new materials. In this context, a recent concept has been proposed for the application of these materials in the development of fingerprints on metallic surfaces, which is of great forensic interest due to the low success rate in the development through conventional methods. This method is based on the insulating characteristic of the sebaceous residue of the fingerprint. The use of electrochemistry makes it possible to manipulate properties and thickness of the conducting polymer films asa function of the applied electrical potential, allowing the adjustment of the visual contrast between the fingerprint and the metal surface [1,2]. Polypyrrole films were electrodeposited on stainless steel surfaces containing latent fingerprints from monomer solutions in aqueous medium, to obtain polymer films with different thicknesses. Electrochemical deposition was performed using a three-electrode system, through different electrochemical techniques in which the experimental conditions (current or electric potential applied, time of application and concentration of the solutions) were varied. The images obtained after the development of the fingerprints were analyzed using the Griaule Forensic Fingerprint software version 1.1, where it was possible to verify the characteristics of a fingerprint such as singular points (core and deltas) and individualizing minutiae (morphology, continuity of ridges, distortions and pores). Therefore, the results of the analysis showed that the images obtained are of high quality for forensic examinations and attend the requirements for the application of technical procedures for human identification.

Acknowledgments:

CNPq, CAPES, FAPEAL, INC Federal Police.

References

[1] Assis AML et al. Perícia Federal v. 40, p. 10-14 (2017).

[2] Assis AML et al. Revista Brasileira de Ciências Policiais v. 11, p. 49-67 (2020).

Electrowetting on Langmuir-Blodgett films from epicuticular wax

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Langmuir and Langmuir-Blodgett (LB) films were fabricated from epicuticular wax, that had been extracted from Vitis vinifera. For Langmuir films, the influence of subphase temperature on their structure was examined, whereas for LB films, the surface morphology at various temperatures and the effect of the layer number on electrowetting were investigated. Isotherms of surface pressure revealed no well-defined phase transition or collapse on Langmuir films at 22 °C, whereas Brewster angle microscopy showed that even before starting the compression procedure, there are platelets of epicuticular wax floating on the subphase. Those platelets give rise to the Langmuir films of epicuticular wax during compression. In contrast, for higher temperatures (40 °C and 50 °C), a well-defined phase transition was found - without epicuticular wax platelets, with a behavior close to those found in typical fatty acids. For LB films, atomic force microscopy images indicated a decreasing of roughness as a function of the subphase temperature as a result of greater organization of the epicuticular wax film. A significant modulation of about 20 ° (under applied voltage range of 0 to 100 V) was demonstrated of the electrowetting on LB films, which did not depend on the LB layer number. Our results suggest that epicuticular wax LB films have the potential for being applied in electrowetting-based devices. Furthermore, the Langmuir-Blodgett technique is feasible to fabricate films with various compositions focusing on the electrowetting.

Acknowledgements: CAPES and CNPq

References:
[1] Oliveira et al., Thin Solid Films, v. 713, p. 138364 (2020).

Evaluation of composite material based on asphaltic concrete and waste foundry sand for road infrastructures

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Composite materials, based on asphaltic binder (AB) and mineral aggregates (MA); aka hot-mix asphalt (HMA); has a worldwide applicability in road infrastructures. However, the currently sustainable development concerns requires sustainable solutions employed on these project considering its high demand of natural resources. In this perspective, the waste foundry sand (WFS) becomes an alternative, due to its high production by foundries and MA similarity. Thus, this paper presents an evaluation on HMA containing 50% of WFS replacing the MA (in weight); according to mechanical, microstructural and chemical properties correlation. The results shown similarity to structural aspects of conventional HMA and within the road engineering international criteria; indicating the technical viability of WFS use. Furthermore, its chemical composition by EDS don't indicatepathogenic elements presence on HMA matrix; considering the WFS residual origin. Inaddition, HMA microstructural analysis by SEM indicated a matrix uniformity composed ofspherical grains, predominantly. Being correlated to its satisfactory resistance to traction (RT), fatigue and resilience (MR) determined by typical road engineering tests.

EVALUATION OF POLYPROPYLENE COMPOSITES WITH POST-CONSUMER GUARANA FIBERS

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The growing concern about environmental impact and the need for sustainable solutions have driven research seeking innovative alternatives for the reuse of waste, such as the residue from the guarana aroma extraction industry. Brazil, one of the world''s largest producers of guarana, generates a significant amount of this waste, making its reuse an opportunity to contribute to sustainability and minimize environmental impact. In this scenario, the search for solutions that integrate this waste into the production of composites, such as the incorporation of natural fibers into a polymer matrix, has gained relevance. This approach aims to combine ecological responsibility with economic efficiency, promoting sustainable development and reducing dependence on non-biodegradable and polluting materials. In this context, the study focused on the production of polypropylene matrix composites using residue from the guarana aroma extraction industry. Guarana fibers were used in two forms: as received and after an alkaline mercerization treatment, which aims to improve the interaction between the fibers and the polymer matrix. The aim of the work was to morphologically analyze the fibers before and after the chemical treatment and to evaluate the mechanical properties of the produced composites. To validate the results, a 2^2 factorial statistical analysis was performed, varyingthe percentage of guarana residue in the composites and the categorical analysis, using thefibers before and after treatment. The response evaluated was the properties of tensile, flexural, and impact strength. The composites were also compared with pure polypropylene. Preliminary results indicate potential in the use of post-consumer guarana fibers in the production of polypropylene matrix composites. If expectations are confirmed, this approachcan offer a sustainable and economic solution for waste management and the production ofrecycled materials.

Exploring Polypyrrole as Extraction Phase for Disposable Pipette Extraction (DPX) Method for Multiclass Organic Micro-Pollutant Determination in River and Tap Water Using Gas Chromatography-MassSpectrometry (GC-MS)

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Concern about the presence of organic micro-pollutants in natural waters is increasing, given the negative impacts on the health of various organisms, including humans [1]. Some classes of these contaminants are pesticides and endocrine disruptor compounds, as personal care products and plasticizers. They are frequently detected in environmental matrices, there is a need for the development of accurate, fast and safe analytical methods for the monitoring of these contaminants in aqueous samples. Thus, sample preparation (which aims to suit the sample to the instrument, preferably pre-concentrating the analytes) is a necessary and very important strategy. Among the sample preparation technique, a microextraction technique have gained a lot of visibility is disposable pipette extraction (DPX) [2]. One of the most positive features of DPX is the possibility of using differentextractive phases. Some of these materials are biosorbents or synthetic materials, like polypyrrole (PPy). The possibility of several interactions between PPy and analytes makesPPy an excellent alternative to be a extractor phase in DPX, is also highly stable, has highsurface area and, especially, is easy to synthesis [3]. This work aims to develop, optimizeand validate a fast and effective method for the determination of 18 organic micro- pollutants of five different classes in environmental waters by DPX using PPy as extractionphase, and GC-MS for separation and detection.

Acknowledgments:
This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior, Brazil (CAPES), finance code 001. The authors are also grateful to the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) for financial support.

References:
[1] Carasek, E.; Merib, J.; Mafra, G.; Spudeit, D.; TrAC, Trends Anal. Chem. 2018, 108, 203.
[2] Pinto, M. A. L.; Queiroz, M. E. C.; Sci. Chromatogr. 2015, 7, 101.
[3] Dogaheh, M. A.; Behzadi, M.; J. Pharm. Anal. 2019, 9, 185.

Extraction and characterization of by-products frompalm bunches

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Oil production in Brazil has been growing year after year. One of the main oils produced in the country is palm oil, popularly known as dendê oil With this increase in palm oil production, there is also an increase in waste generation, particularly the bunches, which are discarded after removing the kernels [1]. In the search for applying these bunches, this research aims to find ways to give new employment for this material. Thus, chemical and physical tests were carried out on the fiber in natura and the extracted cellulose. They also were characterized by X-ray fluorescence (XRF), X-ray diffraction (XRD), and thermogravimetry (TGA) [2]. The results of the chemical analysis showed that the fiber from the palm oil residue presents significant percentages of cellulose (67.2%), being viable for application in composites since this polymer is directly correlated to mechanical properties. The FRX analysis showed potassium (K) as the main element present in the fiber, demonstrating this residue's value for applications in other areas. In the XRD analysis, the characteristic planes of lignocellulosic materials were observed; in addition, crystallinity was increased after its cellulose extraction (32 % to 49%). By TGA, it was possible to observe the thermal stability of the extracted fiber (Tonset= 355 ℃) and the cellulose (Tonset= 370 ℃), showing the possibility of being extruded and injected in a future composite production process. These results show that oil palm bunches can be applied topolymeric composites.

References:
[1] Z. N. Akhlisah, R. Yunus, Z. Z. Abidin, B. Y. Lim, and D. Kania, “Pretreatment methodsfor an effective conversion of oil palm biomass into sugars and high-value chemicals,” Biomass and Bioenergy, vol. 144. Elsevier Ltd, p. 105901, Jan. 2021. doi: 10.1016/j.biombioe.2020.105901.
[2] S. de S. Barros et al., “Waste açaí (Euterpe precatoria Mart.) seeds as a new alternative source of cellulose: Extraction and characterization,”

Fluorescent nanocomposites based on melamine resinand CdTe applied to revelation and sperm marking in latent fingerprints

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The identification and revelation of latent fingerprints (LFP) are standard procedures in criminal investigatio however, currently, the information obtained by this technique can be amplified due to the analysis of markers in the fingerprints, such as biological material [1,2]. In this work, red emitting CdTe quantum dots (QDs) were incorporated under optimum conditions in Melamine-Urea-Formaldehyde (MUF) and Melamine-Thiourea-Formaldehyde (MTF) resins, which were applied as fluorescent powders for simultaneous marking and revelation of spermatozoa in latent fingerprints. The QDs displayed maximum fluorescence in the red region (λ_{ex}= 350 nm / λ_{em}= 590 nm), which was preserved after incorporation in the resin. However, for the MTF/QDs nanocomposite, a higher brightness and fluorescence intensity was found under UV light, possibly associated with the interactions with sulfur groups with Cd atoms of CdTe. LFP was applied in samples without and with semen contamination. The fingerprints revealed details such as pattern type and singular points, indicating high similarity with the donator's fingerprint exploring reference method. Besides, fingerprints with semen contamination were analyzed by optical microscopy, and spermatozoa were found with image resolution with fluorescence emission under UV light. Finally, fluorescent nanocomposites were applied to latent fingermarks deposited on various surfaces (paper, glass, wood, among others) and the marking of spermatozoa; thus, these results suggest that fluorescent powders developed can be used efficiently to help the resolution of sexual crime scenes.

Acknowledgments: LumiAm, LINQA, CNPq, FAPEAL. The present work was supported by the Program of Academic Cooperation in Public Security and Forensic Sciences.

References:

[1] B. Jaqueline et al. Quim. Nova, vol. 42, No. 8, 845-850 (2019)
[2] N. Krishna et al. AIP Conf. Proc., vol 2558, 020047 (2023)

GEOPOLYMER CONCRETE PRODUCTION USINGWASTE FROM KAOLIN MINING

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Geopolymers are inorganic polymers, which are obtained through the polymerization of natural raw materials, of geological origin or by-product materials, containing aluminosilicates [1]. In the production of geopolymers, aluminosilicates are partially dissolved in alkaline solutions containing NaOH or KOH [1]. In geopolymer concrete, geopolymer is used as binder instead of Portland cement. The use of alternative sources of precursor materials is a possible solution for kaolin mining in Pará, which in its mining rocess discards materials rich in aluminosilicates that have no application in the paper industry due to contaminants and characteristics of the ore. The present work carried outchemical, mineralogical and microscopic characterization tests, in order to characterize theore called Hard Kaolin , which is not used as a source of kaolinite for the paper industry dueto technical difficulties in processing, but can be used as a source of aluminosilicates for theproduction of geopolymer concrete. A geopolymeric cement paste was produced and compared with other pastes produced from pure kaolinite and Portland cement, verifying itsmechanical properties through the simple compressive strength test. The results proved that hard kaolin can be an adequate precursor source for this application, and can be an alternative for this environmental liability generated in the kaolin mining activities of the sedimentary deposits of the state of Pará. This generated product also has an ecological,given the origin of its raw materials, minimizing the environmental impacts of miningactivities and taking advantage of a waste that has not yet been used.

Acknowledgements:
To CAPES, PPGCEM, UFPA and Imerys.

[1] CÂNDIDO, V. S., SILVA, R A., SIMANASSIB, T. N. Mechanical and microstructural characterization of Geopolymeric concrete subjected to fatigue. 566-570p. Revista Journal of Materials Research and Technology. 2018.

Homogenous panels produced with sawdust, polyethylene terephthalate and castor oil based polyurethane resin

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The Wood is a raw material widely used in Brazil, and its abundant use in the country can raise concerns about the exploitation of native forests and the generation of waste in the environment. With the constant use of reforestation woods in the production of particleboards, and to reduce the use of resins usual in large panels, and to reduce the use of usual resins in large panel industries, one of the alternatives is desirable in this work, where it partially replaces wood in the addition of Polyethylene Terephthalate (PET). In this study, the present work has to production and evaluation of homogeneous agglomerated panels with shaving from Pinus elliottii, Pinus taeda and PET, bonded with polyurethane derived from castor oil (PUR), with the objective of influence of the addition of PET. Tree blends were presented (two blends containing wood, PET and PUR, and a reference blend with no PET added). For each mixture, tree panels with a nominal density of 0,35 g/cm³ were produced, pressed at a temperature of 160ºC for 6 minutes and a pressure of 70tf. Thepanels were evaluated for their physical end mechanical properties based on the ABNT NBR[1,2] and ASTM [3]. According to the results obtained, the modulus of resistance to staticflexon (MOR) and modulus of elasticity (MOE) were determined, therefore, they were classified as ABNT NBR and ASTM.

References:
[1] Associação Brasileira de Normas Técnicas - ABNT. NBR. 14.810-2: Painéis de partículasde media densidade: parte 2: requisitos e métodos de ensaio. Rio de Janeiro, 2018.
[2] R. A. Bispo, et al. Production and evaluation of particleboards made of coconut fibers, pine, and eucalyptus using bicomponent polyurethane-castor oil resin. BioResources, 2022, v. 17, n. 3, pp. 3944-3951.
[3] ASTM International D1037. Annual Book of ASTM Standard Test Methods for Evaluating Properties of Wood-Base Fiber and Particle Panel Materials. Annual Book of ASTM Standards.

Incorporation of transition metal salt in obtaininghybrid organic-inorganic silicon polymers by hydrosilylation reaction

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The purpose of this study was to prepare hybrid organic-inorganic silicon oxycarbide (SiOC) precursors with different amounts of nickel chloride to evaluate their physical and chemical changes. Polymers were synthed by hydrosilylation reaction between 60 wt.% poly(methylhydrosiloxane) (PHMS) and 40 wt.% divinylbenzene (DVB) containing 20 wt.% polyethylene (PET) and different percentages in mass of $NiCl_2$(0,1,3,5,10 and 20 wt.%), previously dried in the oven. Diffuse reflectance experiments showed that the greater amount of NiCl2 increased the percentage of maximum reflection at 590 nm. TG curves ofNi -free and -containing polymers were like each other, indicating that the presence of NiCl2 did not influence the thermal degradation. The DTG curves showed degradation eventsin 155 ℃ related to H_2O loss for the $NiCl_2$, at 460.6 ℃ (Tmax) characteristic to degradation of matrix PHMS and DVB and at 520 ℃ referent to degradation of PET. FT-IR spectra of thepolymeric matrices indicated that the reaction between DVB and PHMS occurred, due to thedecrease of relative intensities of νSi-H and νC=C absorptions and arising of νSi- $CH_2CH_2$when compared to pure reagents. The polymer crosslinking degree was investigatedby the ratio between νSi-H and νSi-CH_3bands and study of swelling effect test in 10 mL hexane for 24 hours stirring for 0.1 g of each polymers. Both results revealed that the presence of salt favored crosslinking even for 5% wt.% $NiCl_2$material and the materials with10 to 20% the crosslinking was similar to the system without Ni salt. SEM microscopy willbe performed to verify dispersion of $NiCl_2$in polymers. The homogeneous distribution of Ni isdesired since the transition metal associated with a static atmosphere of argon catalyzes theformation of graphite properly dispersed structures implying an application of SiOC material for systems where electrical properties are required.

Acknowledgment
CNPq, CAPES, UEL

Reference

[1]GANGADHAR, J et al. Ceram Int. 2020.

Influence of injection molding parameters on mechanical properties and morphology of short-fiber reinforced polymer composites.

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Energy efficiency has always been one of the main objectives when designing mechanical systems. Fiber-reinforced polymers provide great chemical stability and higher strength-to-weight ratio when compared to traditional metals, hence their wide acceptance in automotive components [1,2]. However, possessing the desirable material is not enough without the appropriate processing method. Injection molding is categorized as an additive manufacturing process which enables the production of complex parts in a short period of time. Therefore, the present study - through the design of experiments (DOE) via Taguchi Method - aims to evaluate the influence of this process on mechanical properties and morphology of such material more specifically on glass-fiber reinforced polyamide with a fiber content of 35 %. The analyzed parameters were injection pressure, temperature, and injection speed. Specimens' density and volume of voids were obtained by Archimedes' Principle; SEM³ allowed the analysis of fiber orientation, and possible voids. DSC³ and tensile testing provided the effect of parameters on the material's crystallinity and mechanical properties such as ultimate tensile strength, Young's modulus and elongation before break. Lastly, using Taguchi Method, such parameters were ranked in order of influence on the aforementioned properties.

[1] R. Autay, S. Missaoui, J. Mars, and F. Dammak, Mechanical and tribological study of short glass fiber-reinforced PA 66, Polymers and Polymer Composites, vol. 27, no. 9. pp. 587-596, 2019.

[2] Recent developments in the manufacturing technologies of composite components and their cost-effectiveness in the automotive industry A review study. Composite Structures. 2021.

Injection Cavity for Pilot Molds Developed in Additive Manufacturing with SLS Technology.

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Industries that seek to develop new products, the faster and more agile this development is, the greater the chance for the industry to place this product on the market. Polymers play a key role in this industrial context, being used in several áreas and the injection molding being one of the most important industrial production processes. However, the negative factor associated with this process is the time and high cost required for tooling development. Additive manufacturing (AM) is currently a prominent production process, with the ability to create parts from a three-dimensional model. In this context, the development of injection mold cavities using AM processes becomes an excellent alternative to assist and speed up the development of tooling required for thermoplastic injection processes. Studies demonstrate that this topic is of great relevance and is already used in some sectors. However, the AM of injection molds using SLS (selective laser sintering) technology is little explored in the scientific and industrial fields. In this work, a hybrid mold was developed using injection cavities produced through AM by SLS technology and machining. The mechanical limits for cavities produced by AM and the durability of cavities produced in Polyamide 12 (PA 12) were evaluated, using polypropylene (PP) test specimens as a reference for injection. The mold was designed to perform the injection of 4 cavities simultaneously, with 2 variations of materials produced by SLS and 2 machined in a metallic material, ensuring that the process parameters are similar for all materials. The test specimens were mechanically characterized by testing tensile strength and flexural strength, as well as dimensional analysis, and the surface of the test specimens was analyzed by scanning electron microscopy (SEM) and thermal analysis. The mold was effective for a pilot model, as it was possible to perform 105 injection cycles, a value that is superior when compared to other authors.

Investigating the Mechanical and Thermal Propertiesof PVC Incorporating Reduced Graphene Oxide Synthesized via Modified Hummers' Method.

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The aim of this study was to synthesize reduced graphene oxide (rGO) from graphite via the modified Hummers' method, and to investigate the incorporation of rGO into flexible Polyvinyl Chloride (PVC) and its resulting properties. Raman spectroscopy and scanning electron microscopy (SEM) were used to analyze the structural, chemical, and morphological properties of the synthesized rGO, which was subsequently incorporated into flexible PVC for polymer characterization tests of mechanical and thermal properties. Graphite was first converted into graphene oxide (GO) using the modified Hummers' method, followed by its reduction to rGO, and incorporation into flexible PVC. The structural, chemical, and morphological properties of both graphite and rGO samples were characterized using Raman spectroscopy and SEM. Polymer characterization tests were conducted to assess the mechanical and thermal properties of PVC with and without rGO. It is expected that the incorporation of rGO will enhance the mechanical and thermal properties of PVC. In particular, the thermal stability of PVC is expected to improve with the addition of rGO, as indicated by the results of thermogravimetric analysis (TGA). Specifically, the thermal decomposition temperature is expected to increase from 180°C to 200°C to 210°C to 215°C. Overall, the incorporation of rGO synthesized from graphite intoPVC has promising potential to contribute to the understanding of the synthesis of rGO viathe modified Hummers' method, and its application in polymer science.

In vitro antimicrobial activity of photo-irradiated safranin-based film-forming solutions as antiseptic in bovine mastitis

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The search for alternative therapies to treat bovine mastitis has been a highly studied topic due to the emergence of multiresistant bacteria [1]. Safranin-O (SFO) is a photosensitizer agent that, when activated by visible light is capable of damaging structures and causing microorganisms death, including Gram-positive bacteria which causes mastitis [2]. This study aimed to prepare in situ film-forming polymeric solutions (FFPS) containing SFO fortopical administration onto cows' teats followed by photoirradiation, to act as antiseptic. The effectiveness of FFPS in inhibiting the proliferation of standard strains of *Staphylococcus aureus*was evaluated. The FFPS were prepared using pure poly(vinyl alcohol) (PVA; Mw = 13.000 to 23.000 Da; hydrolyzed grade = 87 to 89%) at 18% w/w or PVA blended with poly(acrylic acid sodium salt) (PAAS; Mw = 2.100 Da) at 20% w/w. The SFO was incorporated at a concentration of 0.84% w/w. The antimicrobial activity was evaluated by *in vitro*method, with and without exposure to a green LED light (520 nm) for either 1 or 5 min. Both the pure SFO solution (0.84% w/w) and the PVA film were unable to inhibit bacterial proliferation, with and without photoirradiation and, at both times. It was also observed that the photo-irradiated FFPS based on PVA and SFO were able to inhibit the strains of*S. aureus*from proliferation, proving their efficacy. The photo-irradiated FFPS based on the PVA and PASS blend, with and without the presence of SFO, were also able to inhibit bacterial proliferation, suggesting that the photo-irradiated PAAS has antimicrobial properties. The FFPS containing SFO showed promising results for antiseptic use and the antimicrobial activity of photo-irradiated FFPS based on PAAS will be further studied.

Acknowledgements: FAPES - Edital 03/2021; TO 427/2021.

References:

[1] TAKSANDE, J. B., et al. Appl. Pharm. Res., v. 4, n. 3, p. 8-15, 2016.

[2] SILVA-JUNIOR, R. C. et al. ACS Appl. Bio Mater., v. 4, n. 1, p. 581-596, 2020.

Magnetized polyaniline as a reusable adsorbent for the removal of methylene blue dye from wastewater

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The textile industry''s significant advances have caused serious environmental problems since they generate a considerable amount of effluent contaminated with dyes, becoming a concern for governments and environmental protection agencies. This study aimed to obtain an aptmized reusable adsorbent composite (CP) based on polyaniline (PANI) conductive polymer with magnetite (PANI/Fe3O4) for the removal of methylene blue (MB) dye in aqueous solution. The materials were characterized and optimized concerning pH, temperature, and adsorbent concentration. Additionally, an adsorption study was carried out to understand the chemical processes involved, and the reuse of the adsorbent materialwas tested. The results showed that the composites present favorable physicochemical properties for dye adsorption, and the CP 50:1 presented the best performance. Kinetics and equilibrium studies showed that the adsorption process can be described by the pseudo-second-order kinetic and Langmuir isotherm models, respectively. The proposed methodology proved to be efficient for obtaining low-cost adsorbents with the possibility of recovery and reuse in the MB dye adsorption process.

References:

[1] Li, Y., Li, X., Li, J., Li, Z., Sun, S. (2021). Facile synthesis of PANI-Fe3O4 composites and their application for enhanced adsorption of Congo Red. Journal of Materials Science, 56(3), 2311-2326. Bajpai, R., Shukla, M., Shukla, P., Pandey, S. (2021).

[2] Adsorptive removal of acid and basic dyes using PANI-Fe3O4 magnetic nanocomposite: Kinetics and isotherms. Environmental Technology Innovation, 22, 101369. Zhang, J., Li, J., Xiang, Y., Liu, J., Wang, X. (2020).

[3] Preparation of PANI/Fe3O4 nanoparticles by emulsion polymerization and their magnetic and electromagnetic properties. Journal of Magnetism and Magnetic Materials, 501, 166376.

Morphology and composition of thick environmental barrier coating of Al2O3 + SiO2 + ZrO2 from zircon/alumina hybrid precursor using high-velocityplasma spray

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Extreme aerothermodynamic environmental condition arises in hypersonic flight, reentry space vehicles, and modern gas turbines. Inconel superalloys have good oxidation and erosion resistance but has limited operational temperature (850 oC) [1]. Composite C/C, commonly used in aircraft structures due to its low weight and cost, degrade rapidly in a high catalytic oxidizing environment, resulting in loss of its mechanical and structural properties [2]. The solution for such a defying ambient is to place a protective barrier, known as environmental barrier coating (EBC), these coatings are used when the ambient is extremely reactive, with high temperatures and ablatives environment. Next generation EBCs will require stable oxide phases formation, high density, homogeneity, thermal shock resistance, fracture resistance and high thermal conductivity. A worthy candidate, mullite (3Al2O3.2SiO2), is suitable to form composite material and has high chemical stability. In these context thick Environmental Barrier Coatings (EBC) were deposited in carbon-carbon composite using hybrid precursors of alumina and zircon through the high-velocity plasma spray technique [3]. The hybrid precursor consists of a solid dispersed in a solution, both contributing to the coating formation. The EBC's were evaluated in microstructure and composition by the following characterization methods was used, namely DRX, FTIR, Raman spectroscopy, and MEV.

Aknowledgements:
Funding by CAPES 88887.603350/2021-00 Academic Cooperation Program - Defense

References:

[1] Rita, C. C. P., Miranda, F. de S., Prado, E. S. P. Camargo, J. R., Rita, F. L. P., & Petraconi Filho, G. (2021). Brazilian Journal of Development, 7(9).

[2] Miranda, F. S., Caliari, F. R., Campos, T. M., Leite, D. M. G., Pessoa, R. S., Essiptchouk, A. M., Petraconi, G. (2020). Surface and Coatings Technology, v. 404.

[3] Miranda, F. de S., Petraconi, G., Essiptchouk, A. M., Campos, T. M., & Caliari, F. R. (2017). Ceramics International, 43(18).

Multifunctional hybrid films from sudan III and multiwalled carbon nanotubes: electrical conductionand photoinduced molecular orientation

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Multifunctional materials (MFMs) can be an important system because of their capacity to combine different properties useful for electronic devices [1]. Multiwalled carbon nanotubes (MWCNTs) and azobenzene derivate Sudan III (SIII) are compounds with great potential to form this type of system. These compounds exhibit properties of electrical conduction and photoinduced molecular orientation, respectively, which can be combined in a single film-shaped system. In this study, we report on the fabrication and characterization of multifunctional hybrid films created from SIII mixed with MWCNTs via both Langmuir and Langmuir-Blodgett (LB) techniques. For SIII+MWCNTs monolayers obtained using the Langmuir technique, the surface pressure isotherms revealed that the aggregation process of pure SIII drives the formation of monolayers. Brewster angle microscopy of the monolayers indicated that MWCNTs form loop-shaped structures resembling rings. Such structures were not present in films of pure MWCNTs or pure SIII. The current-voltage characteristic curve showed that the films have an ohmic behavior associated to free electrons in MWCNTs, exhibiting conductivity on the order of that of semiconductors. The SIII+MWCNTs LB films exhibited a photoinduced molecular orientation determined via SIII photoisomerization. These processes, occurring in a single film, are the conceptual proof that these systems are MFMs with potential applications in developing hybrid devices.

Acknowledgements: CAPES, CNPq

Reference:

[1] Sousa et al., Thin Solid Films, v. 752, p. 139248-139248, 2022.

Multiple regression analysis of glycerol addition to Cupriavidus necator culture medium on the productionof poly(3-hydroxybutyrate)

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The study aimed to investigate the relationship between the production of P(3HB) (a biopolymer with diverse applications) and four independent variables: glycerol, inverted sugar, temperature, and the concentration of Xt in the culture medium. P(3HB) is a valuable biopolymer known for its potential in medical, environmental, and packaging industries. Multiple regression analysis was conducted using data obtained from a published article [1] to analyze the impact of these variables on P(3HB) production.

The results revealed a strong positive correlation ($R=0.896$) between the dependent and independent variables. The included independent variables accounted for 80.3% of the variability in P(3HB) production, as indicated by the coefficient of determination ($R^2=0.803$). The model was statistically significant ($F=12.231$) with a significance level of 95% ($p<0.00034$), indicating that the regression model adequately captured the relationship between the variables.

Further analysis demonstrated that only the concentration of Xt in the culture medium had a significant effect on P(3HB) production, with a p-value less than 0.05. This finding suggests that increasing the concentration of Xt in the culture medium leads to a corresponding increase in P(3HB) production. However, there was insufficient evidence to suggest significant effects of glycerol, inverted sugar, and temperature on P(3HB) production.

In summary, this study emphas the significance of Xt concentration in the culture medium for maximizing P(3HB) production. By understanding the relationships between the independent variables and the dependent variable, researchers and industries can optimize the production process and harness the potential of P(3HB) in various applications.

References:
[1] Apati GP, Kelbert M, Sombrio BR, Schneider AL dos S, Garcia MCF, Furigo A, et al.. "Avaliação da adição de glicerol ao meio de cultura Cupriavidus necator sobre a produçãode poli(3-hidroxibutirato)." Matéria (Rio J) [Internet]. 2017;22. Available at: https://doi.org/10.1590/S1517-707620170005.0243

Obtainment and characterisation of microcapsules containing ethanolic extract of *Uncaria tomentosa* by spray-drying

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The Uncaria tomentosa has been widely used in traditional medicine, decoctions made from its bark are used for the treatment of various diseases, being the anti-inflammatory activity described as the main pharmacological application, however despite the great benefit its use in the nutraceutical industry is still restricted due to low stability [1]. Thus, for its use is important the application of techniques with protective effect. In this sense, the present work aimed to develop starch microcapsules by spray drying containing macerated ethanolic extract of U. tomentosa bark powder. The ethanolic extract and microcapsule were submitted to chemical and physicochemical characterization and antibacterial activity assays. The results of the characterization of the extract and microcapsules reveal the presence of alkaloids (Dragndorff test) and phenolic compounds (UPLC-DAD). The formation of microcapsule composition was observed by SEM and FTIR. Dissolution tests demonstrated that microcapsules have the characteristic of being poorly soluble in water and the addition of pharmacotechnical adjuvants can aid in the better release and absorption of the active components, reaching ~80% release. The extract and microcapsules presented antioxidant activity with IC50 values of 5.80 and 5.03 μg/mL. The partitioned extracts showed activity against strains of gram-negative and gram-positive bacteria, fraction UT-7 performed best on all strains, reaching MICs below 256 μg/mL, probably dueto the synergistic effect of alkaloids and phenolic compounds. Given the above, the assays performed in this work show the importance of monitoring the quality of Uncaria tomentosa encapsulates to avoid losses by volatilization or decomposition during processing steps and the need for special storage conditions of the extract in solid form.

Acknowledgments: UFAL, IFAL, FAPEAL, CAPES, CNPq, FINEP.

References: [1] M. Sandoval, et al. Free Radical Biology Medicine, v. 29, p. 71-78 (2000).

OBTENÇÃO E CARACTERIZAÇÃO FÍSICO-QUÍMICA DE FILMES À BASE DE BLENDAS DEFÉCULA DE BATATA, QUITOSANA E ALGINATO DE SÓDIO

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O uso de matérias-primas de fontes renováveis vem constituindo um objeto de estudos e pesquisas de diversas instituições, devido ao seu potencial na substituição de produtos derivados do petróleo. Com base nessa afirmação, o objetivo deste estudo foi obter e caracterizar filmes biodegradáveis constituídos por blendas ternárias de biopolímeros. Os filmes produzidos foram compostos por quitosana, fécula de batata e alginato de sódio, combase em um planejamento experimental empregando plano ternário de mistura. As soluçõesfilmogênicas foram obtidas pela técnica casting, com a matéria seca fixa em 2%. O plastificante utilizado foi o glicerol e sua porcentagem foi fixa em 20%, em relação à massaseca de biopolímero. Após a produção dos biofilmes, estes foram cortados em corpos deprova, para a realização da caracterização físico-química, MEV, solubilidade, PVA, cor eopacidade. Foi realizada uma análise de variância para obtenção dos modelos e o Teste Fpara verificar a significância estatística destes. Após essa verificação, foram gerados os gráficos de superfícies de respostas. A partir das superfícies e dos modelos obtidos foi possível avaliar o efeito da composição das blendas biopoliméricas sobre os parâmetros desolubilidade e permeabilidade ao vapor de água. As morfologias das superfícies dos filmesforam analisadas através das micrografias obtidas (MEV). A concentraçãode quitosana naformulação dos filmes influenciou negativamente a propriedade de solubilidade, visto que aquitasana é cerca de 15% menos solúvel e isso ocorre devido a presença de grupamentosamino livres, sendo dessa forma insolúvel em água. Os filmes que continham a presença dafécula de batata obtiveram melhores propriedades de barreira, em relação a blenda que apresentou a menor taxa de permeabilidade ao vapor de água, apresentando ser cerca de15% menos permeável. A partir da morfologia de superfície analisada foi vista a formação compacta e homogênea.

Optimization of self-compacting soil-cement for the manufacture of bricks with the incorporation marble powder and construction waste.

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The objective of this study was to evaluate the influence of marble powder and construction waste on the behavior of self-compacting cementitious soil (SCCS) for brick manufacturing. The parameters of soil, cement, water, and additive were adopted based on recent studies on SCCS. An optimization study was carried out to determine the appropriate proportions of marble powder and waste for brick production. In the fresh state, spread tests in the free regime and confined flow tests from the funnel were performed, while in the hardened state, cylindrical specimens were produced and subjected to axial compressive strength tests at 7, 14, and 28 days and absorption tests. In addition, bricks were produced and tested for compressive strength and absorption. The results showed that the proportion of marble powder and construction waste contributed to better workability conditions and control over flow and segregation in the fresh state. In the hardened state, the best mechanical performances were achieved with 20% marble powder and 10% construction waste, which were adopted for the bricks. The produced bricks presented mechanical strength results around 6.4 MPa and 19.7% absorption. The results indicated that the use of marble powder and construction waste is a viable alternative for manufacturing bricks with satisfactory mechanical and absorption properties.

Acknowledgements:

I would like to acknowledge the Coordination for the Improvement of Higher Education Personnel (CAPES) in Brazil for their support in making this work possible.

References:

[1] R. A. Bispo, G. O. Vicente, G. P. Silva Junior, D. U. Benjamim, M. A. M. Alcântara. Investigation of rheological behavior of self-compacting and high performance composite concretes. Materials Research. 24 (suppl. 2), 2021.

[2] M. Sutcu, H. Alptekin, E. Erdogmus, Y. ER, O. Gencel. Characteristics of fired clay bricks with waste marble powder addition as building materials. Construction and Building Materials, 82, pp. 1-8, 2015.

Permeability of bioplastic films based on starch filledwith graphene oxide

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In view of the different possibilities of using synthetic polymers, they have been mass-produced since the 1940s and, since then, they have been used in the manufacture of several industrial products for everyday use. However, the intensive use of non-biodegradable polymers has caused environmental problems all over the world, such as the contamination of terrestrial and marine ecosystems. [1,2]. Gradually, the plastic waste disposal strategy by industries has been changing. Instead of incinerating plastic waste, which releases toxic products into the environment, government policies have been encouraging the use of biodegradable polymers and creating policies to encourage recycling and composting [3]. The aim of this work was the fabrication of an impermeable and biodegradable starch-based bionanocomposite film filled with graphene oxide to increase moisture resistance, mechanical and thermal stability. Chemical characterization was performed by Fourier transform infrared and Raman spectroscopies, thermal stability and moisture resistance was evaluated by thermogravimetry and contact angle measurements, respectively. Morphology was observed by scanning electron microscopy images. The results obtained in this work showed a reduction in the water absorption of the starch films when 0.1 wt.% of graphene oxide was incorporated in the formulation turning this film a good candidate for application as an intermediate layer in packages based on multilayer films, reducing the use of synthetic plastics or metals and, consequently, minimizing environmental impacts.

Acknowledgements:
The authors thank FAPERJ for the financial viability so important to the development of this work

References:
[1] MALI; GROSSMA YAMASHITA, Agricultural Sciences, v. 31, p. 137 (2010)
[2] GEYER; JAMBECK; LAW, Science Advances, v. 3, p. e1700782 (2017)
[3] https://www.epa.gov/trash-free-waters, accessed in 27/04/2023.

PET/graphene nanocomposites: A study at low concentrations on the electrical conductivity and mechanical properties

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Electrical conductivity and structural durability are essential for their versatile applications of polymer nanocomposites such as electromagnetic radiation absorption, conductive adhesives, and sensors. The incorporation of conductive nanostructures such as graphene isa promising method for conferring conductivity to polymers. Graphene''s high aspect ratio, conductivity, specific surface area, and mechanical strength have made it an excellent choice for the preparation of conductive polymer composites. Understanding the relationship between structure/morphology, processing and final properties is essential for the design and production of materials with appropriate properties for the various emerging applications. In this context, this work investigates the morphology and the transport of electric charges between poly (ethylene terephthalate) (PET) and two types of commercial graphenes incorporated through the melt mixing technique. The results indicate that produced nanocomposites exhibit some type of variation in electrical conductivity, however, there was no significant improvement in the electrical conductivity of the PET/graphene nanocomposites with concentrations ranging from 0.001 to 1 phr. While the other PET samples with exfoliated graphene of smaller lateral size indicated an increase of 44 % in Young''s modulus in relation to pure PET. A relevant result of this study is that for the optimal concentration of 0.1 phr, the use of the smallest lateral size graphene promotes an improvement in mechanical properties in relation to the Young''s modulus.

Acknowledgments:
The authors are thankful for CAPES/PDPG support

Photolysis of aromatic polyamides fiber in Proximity Firefighting Protective Clothing.

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Proximity Firefighting Protective (PFP) Clothing is part of firefighters' personal protective equipment and are developed from fiber blends of aromatic polyamides (Nomex®, Kevlar®and carbon). The integrity of this material is extremely important because it allows firefighters′ thermal and physical protection. On the other hand, PFPs are impregnated with toxic compounds such as PAH and can serve as a source of contamination for firefighters - so they need to be periodically decontaminated properly. One of the ways of decontamination can be through photolysis. Fibers exposure to radiation is a concern, as it can triggers radical reactions promoting material degradation by photolysis reactions[1]. Considering that, we promoted a 3-day and 30-day photolysis PFPC fabric (Unishell®, 58% kevlar®, 40% nomex®, 2% carbon and Teflon® coating) using a light radiation (λ = 283,322, 353, 465, 496, 498, 545 and 577 nm). The fibers′ damage was verified by attenuatedtotal reflection Fourier transform infrared spectroscopy (FTIR/RTA) and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS). Also, a tear resistance test was performed after a 30-day experiment. The FTIR/RTA showed no formation forming acids, alcohols and/or amines, know photolysis by-products[2]. In SEM images no differences were observed fibers morphology. The tear resistance test presented a reduction of 17%-26% after 30-days photolysis. This reduction is not necessarily due to fiber degradation but can be related to removal of Teflon®, evidenced by decrease of fluorine inEDS maps. In view of these results, even after the 30-days photolysis, the integrity of aromatic polyamides fibers maintained.

Acknowledgements:
CAPES, CNPQ and FAPDF.

References:

[1]S.Villar-Rodil, et al., Chemistry of Materials, vol. 13, no. 11, 4297-4304(2001).
[2]R.Davis, et al., Polymer Degradation and Stability , vol. 95, no 9, 1642-1654(2010).

Physical hydrogels of chitosan with inorganic nanoparticles of titanium dioxide modified with peroxo groups (TiO_2-OPM)

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Hydrogels are three-dimensional materials that show the ability to absorb large quantities of water without dissolving. These materials can also respond to stimuli such as pH, temperature, biological molecules, and other properties, and can act in the release of drugs depending on internal and external stimuli, and thus can be classified as intelligent biomedical materials.

Among the polymeric hydrogels, those based on chitosan present interesting characteristics for biomedical applications, such as biocompatibility, gel-forming ability, non-toxicity, and biodegradability. However, some polymeric hydrogels can show poor mechanical properties when compared to other materials, which would limit their applications in specific areas. The incorporation of inorganic nanoparticles into the biopolymer-hydrogels matrix can increase the mechanical and biological properties of the final nanocomposites.

A series of chitosan hydrogels and TiO_2-OPM nanoparticles prepared via an aqueous formulation, only in the presence of ammonia vapor, which induces the gelation process. To compare the effect of peroxo groups on the surface of nanomaterials, TiO_2-OPM nanoparticles were heated at 250°C for 30 min under H_2, generating materials with reduced surfaces free of peroxo groups.

Different factors that affect the properties of hydrogels were investigated through structural, thermal, spectroscopic, and optical characterization. In order to use the material as a vehicle for the controlled release of drugs, the final hydrogels were submitted to loading and release tests with dyes. Results showed that nanomaterials improved the structural properties of hydrogels and showed potential use for drug delivery.

ACKNOWLEDGEMENTS

This work was partly funded by FAPESP (2013/07296-2) and (2022/10016-0), CNPq (309711/2019-3) and CAPES(88887.641038/2021-00), (88887.643328/2021-00) and (8887.828064/2023-00).

PLA/ bulgur amphiphilic films for water and oilabsorption

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An oil spill on roads can be a hazardous situation that needs to be dealt with promptly to avoid accidents and prevent environmental damage. The use of absorbent materials like sand, sawdust, or kitty litter to soak up the oil has been considered. These materials will absorb the oil and make it easier to clean up. It is essential to take immediate action when an oil spill occurs on roads to minimize the damage and prevent accidents. In the present work, films of bulgur [1] and poly(lactic acid) (PLA) (PLA/Bul) were produced to achieve an amphiphilic material. Bulgur (also spelled burghul) is a grain made from whole wheat, and PLA is a biodegradable and compostable polymer [2] that is produced from renewable resources such as cornstarch, sugarcane, or cassava. While bulgur and PLA are organic materials, they are different in their composition and properties, allowing the production of amphiphilic materials. PLA/Bul (with the following composition (PLA: Bul) 90:10; 70:30; 50:50) were produced by compression molding technique and characterized by Scanning electron microscopy (SEM) and FTIR. The film's water and oil absorption (soy oil) were investigated. Composition strongly affected morphology and the amount of water and oil absorbed, as stated by SEM. FTIR indicates that no chemical changes occurred in bulgur and PLA. For 15 minutes, the water absorption of the PLA: Bul films was higher in the samples with the highest amount of bulgur, reaching absorption of approximately 28% wt. However, oil absorption was more expressive in the 70:30 film reaching around 10% wt. These results demonstrate the amphiphilic character of the film associated with the composition.

[1] Ferweez, H., Mohran, M., Tammam, A. A., Ismail, H., Farag, A. New Valley Journal of Agricultural Science, 1, 76, 2021

[2] Rezvani Ghomi, E., Khosravi, F., Saedi Ardahaei, A., Dai, Y., Neisiany, R. E., Foroughi, F., ... Ramakrishna, S. Polymers, 13, 1854, 2021

Polyhydroxybutyrate/Bentonite Composites: Influenceof Bentonite modification in composite´s properties

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Preparation of bionanocomposites, using low percentages of inorganic fillers, is one of the routes to improve some of the properties of biodegradable polymers such as polyhydroxybutirate (PHB). This is because it is environmentally friendly and available in large quantities at relatively low cost [1]. As clays are naturally hydrophilic, in order to make the hydrophilic silicates more compatible with polymers, the cations between the layers can be changed by cationic surfactants like the alkyl ammonium [1,2]. The modified clay becomes organophilic (Bento) and its surface energy decreases and the interbasal distance increases. When the hydrophilic clay (Bent) is rendered hydrophobic (Bento), the interfacial adhesion among the polymer/clay nanocompos-ites phases is enhanced. Furthermore, it makes possible the polymer molecules intercalate inside clays galleries [1,2]. In this work, PHB biocomposites were processed by extrusion in the following PHB/Bentonite wt.%: PHB (100/0); PHB/Bent (90/10); and PHB/Bento (90/10). The samples were characterized by FTIR, SEM, X-ray diffraction, Density, Hardness and Contact angle measurement. New bands corresponding to the alkyl ammonium salt showed intercalation of the quaternary cation in the interlamellar spaces of Bento, according to FTIR/ATR results. DRX diffractogram of Bent indicated the presence of impurities such as quartz, kaolinite and illite; Bento's diffractogram indicated an expansion of the basal layers of the material and that its structure was less crystalline. The density results for formulations PHB, PHB/Bent and PHB/Bento were 1.259 ± 0.03, 1.122 ± 0.04, and 1.337 ± 0.02, confirming a good adhesion with PHB matrix, when organophilic bentonite was used, which corroborate the SEM micrographs.

Acknowledgements:

Faperj and CNPq

References:

[1]R.M.S. Júnior, T.A. de Oliveira,L.M. Araque, T.S. Alves, L.H. de Carvalho, R. Barbosa, J. Mater. Res. Technol. 8(3), 3234-3243 (2019).

[2]A.M., D.P. Mater Sci Eng R Rep 28,1-63 (2000).

Polymethylmethacrylate nanocapsules with damageself-reporting capabilities

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Vitrimers have emerged as a class of polymeric materials with good chemical and mechanical resistance that, in turn, can be reused and recycled. These properties turn vitrimers into an interesting alternative to other polymers in developing a circular economy. Recently, self-healing capacity and recyclability have arisen as some of their desirable characteristics. In this context, the synthesis of polymeric micro or nanocapsules mixed with the vitrimer has emerged. These capsules contain a dye system that is released and activated chemically or physically when the capsule is damaged, showing the place and moment a vitrimer is damaged [1].

The synthesis of polymeric polymethylmethacrylate (PMMA) nanocapsules containing crystal violet lactone (CVL) as a change indicator substance (dye) was studied, employing the emulsification technique. The amounts of PMMA and surfactant, pH and ultrasonic amplitude were varied to evaluate their influence on the and thickness of the capsules. The hydrodynamic was determined by DLS, and the morphology and formation of the capsule were also studied by TEM and SEM. According to our results, 1% is the optimal amount of surfactant, and ultrasonic amplitude plays no critical role in nanoparticle . In addition, the of the nanoparticles increased with the amount of PMMA.

In this work it was developed an experimental design to achieve an optimized preparation process of PMMA/CVL nanocapsules giving rise to a pathway to study their incorporation into a vitrimer model and the demonstration of their functionality. Achieved this, we will be able to advance in the development of the self-repairing capacities of the material and thus contribute to an improvement in the circularity of the economy.

References:

[1] J. Patrick and M. Robb, Nature 540, 363-370 (2016)

Preparation and Characterization of PET/PA6 blend composites with sugarcane bagasse ash as a filler and Joncryl as a chain extender

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This work presents the characterization by torque rheometry, DSC and SEM of PET/PA6 blend composites with sugarcane bagasse ash, synthetized in a Mixer at different mass compositions with some of the compositions having the addition of Joncryl (Jc), a chain extender. The additivated composites presented higher values of stable torque than the ones of the same composition without the extender, however, only the compositions 50%PET/50% PA6 Jc and 100% PA6 presented a positive degradation rate, which means that the chain extension occurred in a way it compensated the degradation during the processing. It was observed through the DSC curve that the polymers, specially the PET, in many of the compositions, presented a higher degree of crystallinity when compared to the neat polymers, indicating that the ash increased the crystallinity, while the additivated composites overall had a decrease in their crystallinity degree, another indication of chain extension. In some of the compositions (75% and 25% PET), the cold crystallization of PET did not occur, which improves its processing and molding, specially through injection stretch blow. All the composites presented a noticeable increase regarding the maximum crystallization rate when compared to the neat polymers, indicating that the sugarcane bagasse ash might have acted as a nucleating agent. At last, although the DSC curve suggested that the blend was immiscible because of the presence of two melting and crystallization peaks, the SEM of the composites showed that the addition of Joncryl appears to have increased the compatibility between polymers.

Preparation of core Shell particles for controlledoxygen release

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Microspheres synthed via the core shell have been used in applications in cells under hypoxic conditions were also demonstrated. The double-layer system allows for the controlled release of oxygen at the optimal level for muscle cell survival. Various approaches have been employed to design and manufacture polymer particles, such as solvent evaporation, emulsion diffusion, nanoprecipitation, microfluidic device, and electrospray. PLA, PCL AND PCO form used microparticles and chloroform as solvent. They are analyzed through optical microscopy and oxygen release test. Resulting in the release of three days in a hypoxic environment and presented spherical morphology where it was possible to observe the formation of the nucleus and the shell. The promising results of this work are translatable to a future regenerative medical application.

Preparation of High Thermal Resistance Polyethylene Nanocomposites with Graphene Oxide for Applicationas Barrier Layer in Oil Pipelines

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High thermal resistance polyethylene (PE-RT) has a unique molecular and crystallinestructure, providing excellent long-term hydrostatic resistance at high temperatures (upto 90 ºC), competing with more expensive engineering polymers. In flexible pipelines,the internal pressure barrier layer must be leakproof. PE-RT is a special type of HDPE with raised temperaturecapability, designed with bimodal resin technology, containing long tie molecules in itscrystalline structure that play a key role in the long-term ductile creep behavior(GRABMANN et al., 2018). It is intended for applications in extreme conditions, suchas aggressive chemicals and hydrocarbons. Graphene oxide sheets (GO) arenanomaterial with numerous applications, mainly due to their high rigidity and gasbarrier properties. It could play an important role as a nanofiller to further improve PE-RT properties. The present work studies the development of PE-RT nanocompositescontaining 0.5, 1.0, and 2 wt.% GO (prepared by oxidation and exfoliation ofcommercial graphite), processed in a twin-screw extruder, and the properties wereevaluated. The results showed that, in all compositions, the addition of nanofillersincreased the thermal stability by up to 13 ºC at the initial degradation temperature ofthe composites, indicating the increase of barrier property. Hardness increased from 50to 59 shore (D) in the composites containing the highest nanofiller content. Thecrystallinity degree increased by 22% for nanocomposites with 0.5% GO comparedwith the pure matrix, and SEM images confirmed good filler dispersion of nanofiller inthe polymeric matrix. GRABMANN, M. K. et al. Aging behavior and lifetime assessment of polyolefin linermaterials. Solar Energy, v. 170, p.988-990, ago. 2018. DOI:10.1016/j.solener.2018.06.046. Disponívelem: am lt;https://www.sciencedirect.com/science/article/abs/pii/S0038092X18305966?via%3Dihub am gt. Acesso em: 17 Mar. 2023.

Production of a voltammetric sensor based on SiOC ceramic material for the simultaneous determination of caffeine, theophylline and peroxidine

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The objective of this work was the production of a voltammetric sensor based on carbon rich SiOC ceramic material for the simultaneous determination of caffeine, theophylline and pyroxidine. The ceramic material was obtained by controlled pyrolysis of a precursor polymer based on the radical reaction between polysiloxane poly(dimethylsiloxane-co-diphenylsiloxane) and Bisphenol A Glycerolate Dimethacrylate, at 1500 °C for 3 hours of annealing. To produce the voltammetric sensor, a paste electrode was produced in the proportions 80:20 (m/m) of ceramic material and nujol® mineral oil, respectively. The simultaneous determination of caffeine, theophylline and pyroxidine is currently little explored and are substances found in large amounts in energy drinks [1]. The ceramic material was characterized by XRD, N2 physiosorption at 77 K and Infrared, Raman and excited photoelectron spectroscopy, which confirmed a relevant amount of conductive (Csp2) and semiconductor (SiC) phases. The voltammetric sensor produced showed a separation of the voltammetric signals, referring to each analyte, more efficient than the commercial glassy carbon electrode and the method for the quantification of the molecules in question is in the finalization process. Furthermore, it is noteworthy that the application of SiOC ceramic materials as electrode materials to produce voltammetric sensors is still little explored, mainly in the simultaneous determination of analytes.

Acknowledgment
CNPq, CAPES, UEL, LADEMA

References

[1] FARAG, Amir Shaaban et al. Electroanalysis, v. 31, n. 8, p. 1494-1499, 2019.

PRODUCTION OF BIOCIDAL POLYMERIC BLENDS BASED ON QUATERNIZED POLYETHYLENEIMINE AND PVA.

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The numerous and structural diversity of pathological microorganisms, along with their mutation capabilities and possibility to cause infectious diseases, shows the need of solutions based on materials capable of inactivating microorganisms on direct contact, that will not be easily overcome by mutations that may be developed [1,2]. Quaternized polyethylenimine (QA-PEI) is promising against a broad spectrum of microorganisms because its quaternary nitrogen atoms can cleave their cell wall or lipoprotein envelope, releasing their genetic material to an extracellular environment [2,3]. In this work, blends of QA-PEI and polyvinyl alcohol (PVA) were produced by solution mixing and drop-casting, producing film samples with QA-PEI proportions of 0%, 1%, 2%, 5%, and 10% in weight into the PVA matrix. Inhibition halo tests performed against both gram-positive S. Aureus and gram-negative E. Coli showed that QA-PEI was effective in inhibiting these microorganisms even at the lowest QA-PEI concentration, as a clear inhibition halo was shown for the PVA/1% QA-PEI sample and their radii increased for both bacteria as QA-PEI concentration increased. TGA analyses showed that the thermal resistance of the samples decreased as QA-PEI concentration raised, although all of the samples only showed significant weight losses starting at temperatures above 300°C. Tensile tests showed a slight increase in the yield point values as the QA-PEI concentration increased, probably due to an electrostatic crosslinking effect between the positive quaternary nitrogen atoms of QA-PEI and the PVA hydroxyl groups. UV-Visible transmittance analyses showed no significant differences between pure PVA and the produced blends.

References:

[1] A. Klibanov, J. Mater. Chem., 17, 2479-2482 (2007).

[2] S. Farah, O. Aviv, N. Laout, S. Ratner, N. Beyth and A. J. Domb, Colloids and Surfaces B: Biointerfaces, 128, 614-619 (2015).

[3] D. Park, J. Wang, A. Klibanov, Biotechnol. Prog., 22, 584–589, (2006).

Production of cellulose acetate membranes modified with graphene oxide and inorganic oxide nanoparticles

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The increasing production of pollutants has negatively impacted the environment and human health [1]. To ensure environmental sustainability, it is essential to prevent the overuse of natural resources and properly dispose of waste [2]. In this context, the use of membranes to separate oil and water emulsions has been a promising technique, mainly to filter extensive effluents. This work studies the application of cellulose acetate membranes modified with graphene oxide and nanoparticles of inorganic oxides for use in the ultrafiltration of oil/water emulsions. The extraction of cellulose from vegetable sponge was followed by the organosolv method in order to obtain the cellulosic pulp [3]. The acetylation of the cellulose obtained was conducted by the homogeneous acetylation method [3]. The analysis of FTIR spectra indicated the disappearance of bands from lignin and hemicellulose. In addition, the spectrum of cellulose acetate showed the characteristic band of the acetyl group and the disappearance of the band corresponding to the cellulose hydroxyls, which indicates the synthesis of the material. The viscosimetric molar mass was determined and the degree of acetylation was obtained chemically, indicating the formationof cellulose triacetate. The produced membranes were then modified with the insertion of graphene oxide and inorganic oxide nanoparticles (e.g., TiO_2), aiming their use for the treatment of oil-in-water emulsions.

Acknowledgments:

This work was supported by CAPES, FAPES, CNPq, FINEP-ANP 53.1 and UFES.

References:

1. ZHU, Y. et al. NPG Asia Mater 6, e101, 2014.
2. TEO, W. et al. International Journal of Biological Macromolecules, 161, 1414, 2020.
3. SANTOS, M. S. et al. Quim. Nova, 43, 279, 2020.

PRODUCTION OF RECYCLED BIODEGRADABLE COMPOUND FROM PAPER AND COCONUT FIBER FOR SEEDLINGS

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Repurposing, repairing or recycling can return materials to active use, creating a technology cycle [1]. Thus, the objective of this work was to produce a biodegradable compound from waste from paper and coconut fiber to be used in the cultivation of seedlings, replacing plastic materials that contribute to the increase in garbage. Paper, coconut fiber (Cocos nucifera) and a gum-based glue were used as raw materials. The biodegradable compound underwent physical-chemical characterization and UV-Vis spectroscopy. The physical-chemical analytical result of the material confirmed the release of elements considered essential for the development of the plant, such as Ca, N, P, K, Mg, Fe, Cu, among others. From the UV-Vis spectra, two significant absorption peaks of 0.81 and 0.87 were identified at wavelengths 257 and 307 nm, respectively. Absorption events in this range probably occurred because it is a lignocellulosic material.

The absorption of radiation is important for plants, however, when subjected to large exposures, it may harm the development of the plant. In general, a biodegradable compound absorbs less. Based on the excellent results obtained, it is concluded that the development of the biodegradable compound contributes significantly to the environment, replacing plastic bags.

Acknowledgements:

The authors are grateful to PNPD/FAPESQ, beneficiary program Renewable Energy, for the financial support.

References:

[1] M. F. Ashby, Chapter 10 - Circular Materials Economics. Materials and Sustainable Development (Second Edition), p. 255-295 (2024).

PVC containing silver nanoparticles with antimicrobial properties effective against SARS-CoV-2

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Poly (vinyl chloride) (PVC) is commonly used to manufacture biomedical devices and hospital components, but it does not present antimicrobial activity enough to prevent biofouling. With the emergence of new microorganisms and viruses, such as Severe Acute Respiratory Syndrome Coronavirus 2 (SARS-CoV-2) that was responsible for the global pandemic caused by Coronavirus Disease 2019 (COVID-19), it is evident the importance of the development of self-disinfectant PVC for hospital environments and medical clinics where infected people remain for a long time. In this contribution, silver nanoparticles (AgNPs) were incorporated into poly(vinyl chloride) (PVC) by melt mixing method. PVC nanocomposites containing Ag/SiO2, Ag/SiO2/ZnO and AgNPs in colloidal suspension (SC-AgNP) were prepared, and their properties were characterized by several techniques as Fourier-transform infrared absorption spectroscopy (FTIR), Thermogravimetric analysis (TGA), Scanning electron microscopy (SEM), Uniaxial tensile tests, Impact tests and antibacterial tests. The results obtained showed the presence of organic compounds in Ag/SiO2/ZnO and SC-AgNP additives. Regarding the morphology of the nanocomposites, there was low adhesion between matrix and reinforcement and a tendency for the Ag/SiO2 and SC-AgNP particles to agglomerate. The best dispersion of the particles occurred in the nanocomposites with Ag/SiO2/ZnO. The mechanical properties of PVC were altered in the presence of the nanoparticles by the low adhesion with the matrix and the influence of the additives on the crystallinity of the polymer. In general, the nanocomposites developed are promising for obtaining materials with antibacterial and mechanical properties.

Study of coating mortars with reuse of rubber wastefrom the tire retreading process

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Abstract: The consumption of non-renewable natural resources in the civil construction industry is an issue that has led researchers to seek sustainable alternatives. The tire retreading process generates waste that can be reused in mortars. Therefore, the objective of this work is to experimentally evaluate coating mortars produced with partial replacement, in volume, of sand by tire retreading rubber (passing the #1.19 mm sieve), in variations of 5% and 10% (AB5 and AB10, respectively), compared to the reference composite, without incorporation of rubber (AB0). For this, consistency, mass density in the fresh state, incorporated air content, mass density in the hardened state and water absorption by capillarity were carried out. The results showed that the incorporation of rubber reduced the consistency index, but did not affect the workability. The mass density, in the fresh and hardened state, also decreased, reaching a reduction of 4.2% for AB10 in relation to the reference, in the hardened state, and this is due to the lower unit mass of rubber in relation to sand and the increase of the entrained air content. The capillary coefficient of composite AB5 remained practically equal to AB0, while for AB10 there was a more significant reduction of 15% in relation to AB0. Therefore, it is concluded that the use of retreading rubber in mortars can be a sustainable alternative in coating mortars.

*Keywords:*Sustainability, Covering mortar, Rubber tire retreading, Civil construction, Reuse of waste.

Acknowledgments

This work was carried out with the support of the Coordination for the Improvement of Higher Education Personnel (CAPES) in Brazil.

Reference

[1] PAGOTO, L.M.; AKASAKI, J.L.; MELGES, J. L. P.; FIORITI, C. F. Analysis of water performance of coating mortars incorporated with tire rubber. Revista Ingeniería de Construcción, 2022, v. 37, p. 58-68.

Study of the electrical conductivity of the HAp–Iron Oxide coatingdeposited by the biomimetic method on Ti–35Nb–7Zr–5Ta alloys

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Abstract

In this study, Ti-35Nb-7Zr-5Ta alloys were coated with hydroxyapatite and iron oxide in order to investigate the electrical conductivity of the samples due to the presence of iron [1]. For this, samples containing hydroxyapatite and iron oxide in different concentrations were prepared. The deposition was performed by the biomimetic method [2]. The samples were characterized by: Scanning Electron Microscopy (SEM), Raman Spectroscopy, Energy Dispersion X-Ray Spectroscopy (EDS), X-Ray Diffraction (DRX) and by the Four Point Probe. Scanning electron microscopy allowed a detailed visualization of the morphology, while X-ray diffraction and Raman spectroscopy were used respectively to study the crystalline structure and chemical composition of the samples [3]. The four-pin probe was used as an essential tool in this study to directly measure the electrical conductivity of the produced samples. The variation in iron concentration in the samples was one of the main objectives of the research, and the four-point probe made it possible to accurately assess the effect of this variation on the electrical conductivity of the samples.

References

[1] T. Kokubo and H. Takadama, *Biomaterials* **27**, 2907 (2006).
[2] D. D. Domingues, *Incorporation of iron oxide into a nanoporous surface of TiO2 grown on Ti10Mo8Nb alloy aiming at biomedical application*, Master's Dissertation, Universidade Estadual Paulista (Unesp), 2019.
[3] L. M. R. de Sousa, M. das Virgens Santana, B. P. da Silva, T. O. Marques, R. R. Peña-Garcia, A. A. Hidalgo, M. L. Vega, B. C. Viana, T. D. Stocco, L. M. R. de Vasconcellos, and others, *Surface Technology and Coatings* **456**, 129249 (2023).

Synthesis and characterization of composite obtainedby combining conjugated polymer poly(p-anisidine) with Au nanoparticles

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Standard chemical oxidative polymerization of p-anisidine was performed. Solution I was prepared by solubilizing 2.0 g of p-anisidine monomer in HCl 1M, and Solution II was obtained by adding 4.0 g of ammonium persulfate also in HCl 1M. Solution II was incorporated dropwise to Solution I under constant stirring. After 3 hours, the dark powder was vacuum filtered, washed, and kept in a desiccator until reached constant weight. For the composite synthesis, the same method as described above was performed to obtain PPA/AuNP but incorporating 10 ml of gold nanoparticle dispersion (AuNP) to Solution I. X-ray powder diffraction was obtained with CuKα radiation, 40 kV, and 20 mA. The scan mode was performed with 0.02°/s step, velocity of 5°/min, in the $2\theta = 3 - 73°$ interval. The XRDpattern of PPA showed a broad halo, between $2\theta = 15°$ and 34°, and centered at $2\theta = 24.5°$, due to the polymer amorphous phase, a strong peak at $2\theta = 5.2o$ and some small, superimposed peaks indicated some crystallinity [1]. Similar pattern was obtained to PPA/AuNP yet exhibiting a smoother halo, indicating some crystallinity improvement. Characteristic XRD patterns corresponding to gold nanoparticles confirm the occurrence ofboth phases in the composite material. With higher crystallinity percentages, we expect toget properties, in general, enhanced for overall applications. Thermogravimetric (TG) analysis already indicates improvement in thermal stability among the composite form.

Acknowledgments
Laboratório Multiusurário de Cristalografia Estrutural (LaMuCrEs)
Grupo de Nanomedicina e Nanotoxicologia (GNano)

References

[1] L. Rodrigues de Oliveira et al., "Head-to-Tail and Head-to-Head Molecular Chains of Poly(p-Anisidine): Combined Experimental and Theoretical Evaluation," 2022, doi: 10.3390/molecules27196326.

Synthesis and characterization of PEO-PPO based polyureas for pharmaceutical drug delivery applications.

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Controlled drug delivery involves the sustained release of bioactive molecules over a given period of time. In this sense, polyurea matrices based on polyetheramine-isocyanates present interesting (physical and chemical) properties for use as drug delivery systems. Polyureas are obtained from the reaction between a polyisocyanate and a polyamine to form urea groups that can interact via hydrogen bonds [1]. To evaluate possible structural changes and thermal stability of the polyurea, polyetheramine-based polyurea networks were synthesised with polypropylene oxide (PPO) or polyethylene oxide (PEO) and hexamethylene diisocyanate-HDI trimer [2]. The network formation was controlled by sol- gel reactions, and the obtained materials were characterised by different techniques (FTIR,XRD, XRD, TGA). Furthermore, it was possible to modulate the amount of diclofenac released as a function of time by studying the water absorption or swelling capacity, cytotoxicity and toxicity. The release profile was then correlated with the water absorptionby the PEO/PPO polyurea matrix (of similar molecular weight), and the release profile wasanalysed with the water absorption by the PEO/PPO polyurea matrix [3]. Finally, links couldbe established between diclofenac release and properties of the polyurea matrices, such aswater absorption capacity. Our aim here is to identify challenges and opportunities for thedevelopment of innovative functional biomaterials for healthcare applications.

Acknowledgements: FAPESP (grants #2021/06552-1, #2022/06507-9, #2022/13408-7 and EMU #2021/14619-9), CAPES (Finance Code 001), and CNPq (grant #307696/2021-9).

References:

[1] R. Heath, Mater. Eighth Ed. (2017) 799-835.

[2] J. Du, N. Ibaseta, P. Guichardon, Chem. Eng. Res. Des. 182 (2022) 256-272.

[3] Y. Shibasaki, R. Kudo, T. Tsukamoto, Y. Oishi, React. Funct. Polym. 176 (2022) 4-11.

Synthesis of a bifunctional hybrid molecularly imprinted polymer for chromatographic detection ofdiuron in environmental and food matrices

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The increased use of the herbicide diuron for weed management is a contributing factor to its environmental release, leading to the contamination of drinking water sources. Given its persistence in the environment and harmful impacts on human health, monitoring both in the environment and in food is crucial.[1] The purpose of this work was to synthe a bifunctional hybrid organic-inorganic molecularly imprinted polymer (BHMIP) by the sol-gel process and free radical polymerization for the quantification of diuron in aqueous and food matrices. The polymer was synthed using methacrylic acid and phenyltrimethoxysilane as organic and inorganic functional monomers, respectively. The BHMIP was characterized by means of MET, SEM, FTIR, X-ray photoelectron spectroscopy, and TG. The printed polymer was used as an adsorbent for solid phase extraction (MISPE), in the development of a chromatographic method (HPLC-DAD) of preconcentration of diuron, whose optimized conditions were: 150.0 mg of mass of PHB-MIP filled in the SPE cartridge, preconcentration volume (pH 4.0) of 50.0 mL and 6.0 mL methanol as eluting solvent. The MISPE method showed a linear range of 0.27 to 200.0 µg L-1, a high pre-concentration factor, evaluated at 173, with a max adsorption capacity of 140.1 mg g-1. The LOD achieved was 0.08 µg L-1. The proposed method was applied to samples of water and soy milk. Samples were enriched with diuron and satisfactory recovery percentages were achieved (91 to 104% and 93 to 110%, respectively). Given the results obtained, using two functional monomers ensured high adsorptive capacity and selectivity to the material. It was also possible to apply it to environmental and food samples without matrix effect.

Acknowledgments: CAPES, CNPq, UEL.

References:

[1] G.F.Alves et al., Journal of Food Composition and Analysis, 106, 104284 (2022).

Synthesis of conductive composite based on bacterial cellulose and polyaniline by an alternative method

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Bacterial cellulose (BC) is a biopolymer, which can be obtained from renewable sources. It is biocompatible and has good mechanical properties. Much researchs has focused on BC for applications biomedicals, food packaging, water purification and conductive composites [1]. Among the conductive polymers, polyaniline (PAni) has gained prominence due to its good electrical conductivity, good chemical stability and ease of synthesis, however, it has low mechanical strength [2]. Therefore, the combination of PAni properties in a CB matrix results in a polymeric film, flexible, porous and conductive. In this work, was used a new route for preparing conductive composites based on polyaniline and bacterial cellulose, obtained by conventional method [3] and an alternative method. Were compared and evaluated through the analysis of conductivity by the four-point probe method, impedance and cyclic voltammetry. The results of DRX, UV-Vis and Raman, indicate that these composites presented structural characteristics, according to the literature. Too, they presented conductivity, identified by means of a multimeter. Therefore, it is believed that these materials can have the potential for applications such as electromagnetic interference shielding flexible electrodes for electrochemical sensor and supercapacitors.

Acknowledgments:
Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) for granting the research grant.

Reference:
[1] S. Choi, K. Rao, S. Zo, E. Shin, S.Han, Polymers, 14, 1080 (2022)
[2] A. Nada, A. Eckstein, J. Mosnácek, International Journal of Molecular Sciences, 23, 842 (2022)
[3] S. Hestrin, M. Scharam, Biochemical Journal, 58, 345 (1957)

Synthesis of lignin-based carbon/graphene oxide foam and its application as anelectrochemical sensors

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Currently the bio-based products industry is ineffective in using lignins as a resource for sustainable and circular materials. Thus, a promising application of kraft lignin (KL) is to use it as a carbon source to produce carbon foams (CF). CF are materials with a pore structure, formed mainly of macro-pores that bond together, generating materials with light relative density and high electrical and thermal conductivity. The additivation with graphene oxide (OG) and its reduced form (rGO) is a promising branch since they are carbon-based nanomaterials with unique physical and chemical properties, such as high surface area and mechanical strength that allow various applications such as supercapacitors, sensors, filters, among others. In this work, the soluble fraction of KL was used as a partial substitute for phenol in phenolic resins, added with 1, 2 and 4% GO and rGO to prepare CF, using polyurethane foams as sacrificial mold. The obtained CF were studied for application in electrochemical sensors. The CF were ground and sieved, and then the powder obtained was adhered to the surface of a conductive substrate. For this, an electrochemical study was performed using the techniques of cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS), to verify the trend of the current density (J) and the values of the charge transfer resistance (Rct). The samples that showed the best J and Rct results were GO2% and rGO2%, which is the best percentage of additivation, however all CF produced showed electrochemical response and potential application as sensors.

Acknowledgments:
This work was financed in part by the Coordination for the Improvement of Higher Education Personnel - Brazil (CAPES) - Financing Code 001. Process Number: 88882.427090/2019-01 (JSR), 88882.430936/2019-01(ASMF) and 88887.497915/2020-00 (CCM), São Paulo State Research Support Foundation-FAPESP (Project 2019/19401-1). This work made use of Aalto University Bioeconomy Facilities.

Synthesis of PMAEMA copolymers for antimicrobial materials

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The synthesis of antimicrobial polymers has attracted great interest lately, particularly for coating of different surfaces in order to avoid the dissemination of viruses and bacteria.[1,2] The objective of this work is to synthe materials via RAFT, seeking to study their antimicrobial performance accordingly to their composition and molar mass.[3] Two syntheses of copolymers were performed by RAFT, varying percentage compositions (50%DMAEMA:50%MMA and 30%DMAEMA:70%MMA). The use of a RDRP technique is mandatory in this kind of approach to guarantee interchain composition homogeneity. Molar mass monitoring was performed by Gel Permeation Chromatography (GPC), demonstratinga kinetic pattern compatible with controlled polymerization for both materials. The copolymer with equimolar percentage proportion presented a slower rate of polymerization at the beginning of the reaction. The two purified materials had polydispersity index below 1.5 and were characterized by FTIR. The materials are to be tested for the antibacterial activity in order to study the influence of PDMAEMA composition.

Acknowledgements

This work was supported by FAPESP, together with the Materials Engineering Department of the Lorena Engineering School of the University of São Paulo.

References

[1] Jain, A.; Duvvuri, L. S.; Farah, S.; Beyth, N.; Domb, A. J.; Khan, W. Antimicrobial Polymers. Adv. Healthcare Mater. 2014, 3 (12), 1969-1985. https://doi.org/10.1002/adhm.201400418.

[2] Alvarez-Paino, M.; Munoz-Bonilla, A.; Fernandez-Garcia, M. Antimicrobial Polymers in the Nano-World. Nanomaterials 2017, 7 (2), 48. https://doi.org/10.3390/nano7020048.

[3] Moad, G.; Rizzardo, E.; Thang, S. H. Living Radical Polymerization by the RAFT Process - A Third Update. Aust. J. Chem. 2012, 65 (8), 985-1076.

The effect of CTB on the production of hybrid material

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Anthocyanins (ACN) make up the largest group of water-soluble pigments in the plant kingdom. However, they have a disadvantage compared to synthetic dyes due to the change in color during the processing and storage of the material. One of the strategies to improve the stability of ACN is the production of hybrid ACN pigments with clay minerals. The present work aims to produce hybrid pigments based on Bentonite (Bent) intercalated with cetyltrimethylammonium bromide (CTAB) and anthocyanin dye (ACN). The hybrid pigments were characterized by X-ray diffraction (XRD), infrared spectroscopy (FTIR), and scanning electron microscopy (SEM).the hybrid pigments based on the natural dye anthocyanin and the clay mineral bentonite were obtained, in which the influence of the amount of CTAB introduced in the previous modification of the clay mineral was evaluated. In addition, DRX, FTIR, and SEM characterization techniques proved that organic molecules were intercalated between the mineral layers of the clay while producing the hybrid pigments

Thermal and mechanical evaluation of adobesreinforced with coconut fibers

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Adobe is an unburned brick to which stabilizers are commonly added to improve its mechanical characteristics. The main objective of this work is to evaluate the impact of coconut fibers addition on the mechanical and thermal performance of the manufactured adobes. To do this, the soil was initially characterized by means of a granulometric test. Then, the morphology of the coconut fibers was evaluated by scanning electron microscopy (SEM). For the structural characterization, soil and fiber samples were subjected to X-ray diffraction (XRD) technique. Furthermore, in order to determine the chemical composition of the raw materials, an evaluation was carried out using the Fourier transform infrared spectroscopy technique (FTIR). After that, it was determined which compositions of adobes would be studied: M1 (reference, without fiber addition), M2 (addition of 0.25% w/w of fibers with 5 cm), M3 (0.50% w/w of fibers with 5 cm), M4 (addition of 0.25% w/w of fibers with 2.5 cm) and M5 (addition of 0.50% w/w of fibers with 2.5 cm). The adobes were also evaluated in terms of their mechanical strength, by means of compression test, and thermally, using the heat flow method. The results of the granulometry define the soil as loamy-sandy-clayey. For the fibers, SEM showed their rough nature. In addition, the experimental powder pattern of the soil confirmed the presence of silicon oxide (SiO_2), reference ICSD 089277, in its composition. XRD analysis of the fibers revealed the semi-crystalline nature of this material. Furthermore, when comparing the strengths of the adobes, better results were obtained for batch M4 (average of 1.89 MPa) and worse for batch M3 (average of 1.31 MPa). In the thermal evaluation, batch M5 stood out, with a thermal damping of 59.39%. Thus, the results showed an improvement in the thermal and mechanical properties of adobes with the addition of coconut fibers.

Acknowledgements:
The authors are thankful to the CAPES and FINEP (ref. 0533/16).

Thermal, optical and morphological properties of PVA/chitosan blend incorporated with parsley extract

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Nowadays, research on active packaging includes the incorporation of natural active substances with antioxidant properties and antimicrobial activity [1]. In this work, a mixture of the equal volumes of the PVA prepared polymer blends of polyvinylalcohol (PVA), chitosan (CH) and parsley extract (PE) and CH solution kept under stirring for 1h. Then, a volume of parsley extract required to produce PVA/CH films with 8 and 16% extract was added. The mixture was transferred to a non-stick surface and taken to the oven for 19 h at ≈50 °C. UV-VIS spectroscopy revealed that the incorporation of PE into the PVA/CH matrix promotedthe shift of the main band of the host matrix. The photoluminescence (PL) of the parsley extract as well as the PVA/CH/PE film, when excited at 450 nm, showed a band in the rangeof 460 to 620 nm with a maximum at 522 nm, while the pure PVA/CH film showed noluminescence in this region of the spectrum. Using a □exc at 300 nm, the parsley extractshowed an emission band at 408 nm, while the PVA/CH/PE film showed an emission with amaximum at 390 nm. At this □exc (300 nm), pure PVA/CH blend showed the same emissionspectrum as the blend incorporated with PE. Thermal analysis showed that parsley extract is easily thermally degraded. The PVA/CH/PE films showed four phases of degradation ThePE extract does not change the thermal stability of the blend. Scanning electron microscopy(SEM) of the surface of the 8% PVA/CH/PE film showed smoothies with small well-scatteredholes. However, micrographs of PVA/CH/PE 16% revealed different morphology with the assigned groove in specific circular regions. The internal morphology of the 8% PVA/CH/PEblend confirmed the existence of circular holes, while the 16% PVA/CH/PE blend had a smooth surface.

Acknowledgments

The authors would like to thank the Federal Institute of Brasilia (IFB) and FAPDF for financial support.

References

[1] Nune J. C; Journal of Food Science and Technology, 58, 1, 2021.

THERMODYNAMIC AND KINETIC STUDY OF RH B ADSORPTION BY BIOPOLYMER INCORPORATED MAGNETITE NANOCOMPOSITES

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The present work studied the synthesis, characterization and functionalization of magnetic nanoparticles and magnetic nanocomposites based on iron oxide functionalized with soybean oil-based biopolymer, (BPLMR), with the purpose of environmental remediation (adsorption studies) of organic compounds in aqueous solution, such as rhodamine B (Rh B) [1]. The biopolymers associated with magnetic nanoparticles have several advantages, such as good surface area [2], magnetic properties that aids in the recovery of the adsorbent, among others. Nanoparticles have been used for the adsorption of organic compounds and metal ions with considered adsorptive efficiency [3]. In this regard, strategies to obtain stabilized nanomaterials in solution is very important as they can improve colloidal stability and surface properties such as adsorption. The synthesis of nanoparticles was be performed by the co-precipitation method and for the characterization of these nanostructures, scanning electron microscopy (SEM) and transmission electron microscopy (TEM), X-ray diffractometry (XRD), Fourier transform infrared spectroscopy (FTIR), kinetic and thermodynamic study and UV/Vis spectroscopy analysis will be used the obtained results will be compared in the literature with functionalized Fe3O4-based nanoparticles and other nanoparticles.

Acknowledgments: UNESP, PROPE, CNPQ

References:[1] TANG, S. C. N. ; LO, I. M. C. Water Research, 47, p. 2613-2632, 2013[2] GAO, F. ; GU, H. The Royal Society of Chemistry, 5, p. 60208-60219, 2015.[3] YANG, J. ; PARK, S.-B. International Journal of Pharmaceutics, 324, p. 185-190, 2006.

Thermo-optical properties of PVA films doped with carbon dots

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Polyvinyl alcohol (PVA) is a water-soluble polymer that presents biocompatibility and biodegradability, being widely used for clinical applications, production of films, fibers, and materials packaging. Moreover, a remarkable amount of attention has been devoted to PVA due to its transparency, film-forming capability, mechanical rigidity and high chemical resistance. Recently, the application potential of this polymer has been demonstrated in electro-optical devices and flexible solar cells. Several studies have been carried out focusing on the doping of PVA films with nanomaterials, in particular, with carbon quantum dots. Some studies have shown that the addition of these nanoparticles improves the mechanical and optical properties of PVA films. In this study, the spectroscopic and photothermal properties of polyvinyl alcohol polymeric films, doped with carbon quantum dots derived from methyl red azo dye, are investigated. The impacts of nanoparticles addition into the polymer matrix are explored, with emphasis on absorption and emission spectra, FTIR and lifetime measurements. Using time-resolved Z-scan measurements, it is investigated how film doping modifies the thermo-optical response of these materials. From the thermal lens model, it is investigated how the concentration of carbon quantum dots affect the thermal diffusivity and the thermo-optical coefficient of PVA films. Thermal effects on fluorescent properties of PVA - CD-MR composite are analyzed.

THE ROLE OF CYSTAMINE DIHYDROCHLORIDE MEDIATING REDOX-SENSITIVE GELATIN NANOHYDROGELS

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Cancer is the second leading cause of death worldwide after cardiovascular disease, according to the World Health Organization [1]. Aside from mortality, the economic impact has grown year after year due to rising medical and hospitalization costs. As a result, new medical devices to reduce or even treat cancer are required. In this study, gelatin was derivatized by maleic anhydride to produce nanohydrogels. With a grafting degree of 45%, the derivatization steps anchored maleimide groups onto the gelatin backbone. A Michael-addition reaction between gelatin maleimide groups and cystamine dihydrochloride amine groups crosslinked the derivatized gelatin. In addition, the hydrophilic and lipophilic balance was adjusted to improve drug uptake and release A disulfide linkage in the cystamine molecule demonstrates sensitive responsiveness to reduced glutathione expressed in cancer cells [2]. Derivatized gelatin was crosslinked in an inverse emulsion system, where particles with hydrodynamic diameters ranging from 200 to 400 nm were obtained. Sodium diclofenac was used as a drug model. The structure of nanohydrogels was evaluated during the release step and the value of hydrodynamic radius and gyration radius were compared as a function time. The gelatin nanohydrogels produced have the potential to be used as a drug delivery system.Acknowledgments:CAPES, CNPq and, IQ UNESP.References:[1]. ORGANIZATION WH. World health statistics 2018: monitoring health for the SDGs, sustainable development goals. Published 2018. Accessed May 22, 2019. https://www.who.int/gho/publications/world_health_statistics/en/[2]. Kennedy L, Sandhu JK, Harper ME, Cuperlovic-culf M. Role of glutathione in cancer: From mechanismsto therapies. Biomolecules. 2020;10(10):1-27. doi:10.3390/biom10101429

Twaron® fabrics impregnated with fluid and shear thickening gels (STF and STG) - applications forballistic vests

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Several studies are being developed to achieve flexibility without compromising the performance of ballistic vests using other materials in conjunction with p-aramid fabrics. The so-called "liquid armor" stands out. This type of armature consists of fluids or gels with non-Newtonian behavior, which, under normal conditions, are flexible. However, the viscosity increases when subjected to mechanical disturbances capable of exceeding a critical shear rate value. The fluid or gel ceases to be liquid/thick and starts to have the characteristics of rigid materials, thus protecting the vest from being penetrated by the projectile[1]. In the present work, composites were produced with three layers of p-aramid fabrics impregnated with shear-thickening fluids and gels (STF and STG), which were prepared based on polyethylene glycol (PEG 200), hydroxylated liquid polybutadiene (PBLH) and nanoparticles of silica ($SiO2$), zinc oxide (ZnO) or boron carbide ($B4C$). It is important to note that synthed gels based on polyurethanes are unprecedented for such applications. The results showed that the thermal cross-linking reaction increased the hydrophobicity of the PBLH gels, confirmed by the contact angle test. The ballistic tests showed that the friction between the yarns increased after the impregnations, changing the failure mode of the fabrics from deformation in pure yarns to fiber breakage in treated fabrics.

Furthermore, it was noted that impregnated fabrics achieved better ballistic resistance through increased impact energy absorption. Comparing STF and STGs of both polymers, it was evidenced that the PBLH performed better. The fabrics impregnated with polyurethane gels with the addition of B4C nanoparticles proved promising for application in ballistic fabrics.

Acknowledgments: CAPE UFRJ-IMA

References: [1] HUANG, W.; WU, Y.; QIU, L.; DONG, C.; DING, J.; LI, D. Advances in Condensed Matter Physics, v. 2015, p. 1-5 (2015).

Use of Carbon Nanofibers in UHMWPE/PEG Blend:Analysis of Morphological and Mechanical Properties

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The preparation of polymeric nanocomposites with fillers has been a way to obtain materials that help to improve the performance of polymeric materials. Nanofillers have emerged with great potential for use in polymeric nanocomposites, mainly due to their mechanical and thermal properties [1]. In this work, polymeric nanocomposites with carbon nanofibers (CNF) were prepared by grinding the powders in order to evaluate the modifications promoted by the incorporation of CNFs in the matrix of a UHMWPE /PEG blend through the morphological and mechanical properties. Grinding was carried out in a pitcher mill with concentrations of 0.5; 1.0 and 1.5% (w/w) CNF for a 24 hour period. All samples were characterized by scanning electron microscopy (SEM) and mechanical tensile tests. Regarding the mechanical properties of the nanocomposites, the CNFs were not efficient, causing a decrease in the properties of the UHMWPE /PEG blend due to the dispersion and distribution of the fibers not being efficient. The SEM images showed the formation of clusters of CNFs in the blend matrix, which caused a decrease in Young's modulus and tensile strength. On the other hand, there was an increase in the ductility of the blend as the amount of CNF in the blend matrix increased [2,3].

Acknowledgmets: This work was supported by CAPES.

References

1. BARRENA, M. I. et al. Applied Surface Science, v. 289, p. 124-128, 15 jan. 2014.
2. PANG, W. et al. RSC Advances, v. 5, n. 77, p. 63063-63072, 2015.
3. SUÑER, S. et al. Composites Part B: Engineering, v. 78, p. 185-191, 1 set. 2015.

Verification of accidental humidity in different wall coatings using infrared thermography

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Abstract: In order to analyze the potential of the thermographic technique, a simulation of real conditions of accidental humidity was carried out in mortar test specimens, differentiated by coatings of ceramic plates, plaster, stone and mortar. Two evaluation and identification techniques were used, with a common parameter: temperature. The techniques, passive and active, are extreme in the induction or not of heat transfer to the prototypes, monitoring in a methodological process of time, temperature and emissivity with the resource of capturing images of visible light with the thermographic camera. Based on the development of the study, the results and discussions were partial to what was expected, since the object of study being at a higher temperature, that is, during the active test, the temperature difference was more clearly visible, which facilitated the detection of moisture, however, in coatings such as natural stone and ceramic tiles, the techniques were not very effective. Therefore, the color gradient has a positive influence on the final result and all the necessary procedures and parameters, in addition to physical characteristics, directly influence the achievement of results, based, in particular, on identifying the pathological manifestation studied, differentiating the results by method passive and active.

*Keywords:*Anomalies, Thermographic technique, Accidental humidity, Coatings, Nondestructive method

Acknowledgments

This work was supported by PIBIC/CNPq.

Reference

[1] ALAM, F. W.; PINZ, F. P.; TORRES, A. S.; PALIGA, C. M. Potencial da termografia infravermelha na detecção de manifestações patológicas ocultas ocasionadas pela umidade acidental em paredes de alvenarias cerâmicas com revestimento de argamassa e pintura. Revista de Arquitetura IMED, 5(1): 28-47, 2016.

D-Advanced Materials Applied in sensors for Health, Agricultural, and Environmental Applications

Gas sensors employing nanocrystal/cluster modified graphene

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The release of harmful gases into the atmosphere, is becoming a major societal concern due to its effects on human health and the environment. While graphene has emerged as a promising material for developing room temperature operated gas sensors, its inertness and lack of specificity results in low sensitive and poorly selective devices. This lecture addresses several approaches for overcoming the drawbacks mentioned. These consist of loading graphene with polymer nanoparticles, metal clusters or perovskite nanocrystals, thus tuning their gas sensing properties. The synthesis, characterization, sensing properties and sensing mechanisms will be presented and discussed in detail. Additionally, reliability and long-term stability aspects will be dealt with as well.

Acknowledgements:

Funded in part by Marie Skłodowska-Curie Actions (MSCA) no. H2020-MSCA- RISE-2018-823895 “SENSOFT”, E.L. is supported by the Catalan Institute for advancedstudies (ICREA) via the 2018 Edition of the ICREA Academia Award.

References:

[1] J. Casanova-Chafer, P. Umek, S. Acosta, C. Bittencourt, E. Llobet Title: Graphene Loading with Polypyrrole Nanoparticles for Trace-Level Detection of Ammonia at Room Temperature, ACS Appl. Mater. Interfaces, 13, 34, 40909-40921.

[2] J. Casanova-Chafer, R. Garcia-Aboal, P. Atienzar, M. Feliz, and E. Llobet, Title: Octahedral Molybdenum Iodide Clusters Supported on Graphene for Resistive and Optical Gas Sensing, ACS Appl. Mater. Interfaces 2022, 14, 51, 57122-57132.

[3] Casanova-Chafer, J.; Garcia-Aboal, R.; Atienzar, P.; Llobet, E., Title: Unraveling the Gas-Sensing Mechanisms of Lead-Free Perovskites Supported on Graphene, ACS Sensors 7(12), pp. 3753-3763

The role of surface oxygen vacancies in the gas sensing mechanism of SnO2

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Conductometric gas sensors (CGS) provide a reproducible gas response at a low cost but their operation mechanisms are still not fully understood. Using synchrotron radiation, we investigated a working SnO_2sensor under operando conditions via near-ambient pressure (NAP) XPS with simultaneous resistance measurements, and created a depth profile of the variable near-surface stoichiometry of SnO_{2-x}as a function of O_2pressure. Our results reveala correlation between the dynamically changing surface oxygen vacancies and the resistance response in SnO_2-based CGS. While oxygen adsorbates were observed in this study we conclude that these are an intermediary in oxygen transport between the gas phase and the lattice, and that surface oxygen vacancies, not the observed oxygen adsorbates, are central to response generation in SnO_2-based gas sensors.

Wearable sensors and biosensors for monitoring health and food quality

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The concepts of personalized medicine and precision agriculture have gained momentum with a number of technologies based on wearable sensors and biosensors that can be used to monitor health conditions, and water and food quality at the point of use. Because the principles of detection and the synergy achieved in combining nanomaterials and biomolecules are common to all of these technologies, a single platform can be establishedin spite of the variety of materials employed and of the distinct targeted applications. In this lecture, examples will be given of wearable electrochemical biosensors to monitor the concentrations of urea in sweat and of glucose in urine. In both cases, the cost of each sensing unit is very low and the measurements are performed with portable devices, thus allowing for point-of-care diagnosis. Also discussed will be electrochemical paper sensors for non-destructive routine analysis of pesticides on plants, fruits, and vegetables, which can be performed over the entire food processing chain, from the farms up to the industry and supermarkets. A key feature in this sensing platform is the ability to control the molecular architecture in the biosensors which are made essentially of layered nanometric films. The large of amount data generated while monitoring the different markers and contaminants are now routinely analyzed using machine learning approaches, with which efficient intelligent systems can be built.

Working towards sensing at room temperature with carbon nanotubes

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The electronic and optical properties of carbon nanotubes are very sensitive to the chemical environments. It is therefore inferable why they are so attractive as sensing objects. These one-dimensional nanostructured materials have been the focus of intensive research but it is important to keep in mind that practical difficulties to produce them ultra-clean and nearly defect-free have constantly appeared as stumbling blocks for their use in applications controllably and reproducibly. One of our pilot experiments -to understand how the intrinsic properties of single-walled carbon nanotubes vary when they are exposed to gases- was to investigate the adsorption of O_2, NO and NO_2molecules. We performed experiments on ultra-pure nanotube material and then tested the influence of molecular fillers. Strikingly, we figured out that many of the sensing effects that had been reported previously were produced in great part by the presence of impurities and defects. This affects tremendously the reproducibility of the experiments. We have also shed light on the nature of adsorption and its effect on their electronic properties of the tubes. Metallicity-sorting and the further use of carbon nanotube hybrids are able to unfold two major challenges: tuning the gas-tube interaction and achieving desorption of gases at ambient temperature. A result to highlight was that using time resolved photoemission we observed that in semiconducting nanotube hosts the chemical potential is pinned inside their energy gap shifted to the onset of the conduction band when nanotubes filled with nanoclusters are used. This remarkably reveals that having a metal cluster filling could represent a key to high sensitivity with a very effective desorption at ambient temperature. I will present an overview of my group's related research.

Affordable Materials and Methods for Acoustofluidic Device Fabrication

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Acoustofluidic devices are becoming increasingly popular due to their precision and efficiency in manipulating fluids and particles at micro/nanoscale. However, their widespread use is limited by the high cost of traditional fabrication methods. In this talk, we introduce a new approach for the fabrication of acoustofluidic devices using affordable materials and methods such as 3D printing, laser cutting of glass, and double-sided tape. We showcase the feasibility of this approach by creating a range of devices, including standing-wave resonant devices. Our devices have potential applications in medical diagnosis and live-cell monitoring. For example, using our standing-wave device, we were able to perform immunoagglutination tests with high precision and accuracy for various analytes, including bacteria, viruses, and proteins. This is important for early disease diagnosis, where low concentrations of analytes must be detected in complex biological samples. Additionally, we can use our devices for live-cell Raman spectroscopy, which provides valuable information about the chemical composition of living cells. Unlike conventional Raman methods, our approach enables non-contact and non-invasive manipulation and trapping of individual cells, allowing high-sensitivity and high-selectivity live-cell Raman spectroscopy. This is particularly important for studying cellular processes and the effects of external stimuli on cell metabolism. Our approach provides a low-cost and easy-to-implement solution for fabricating acoustofluidic devices with high precision and accuracy. This makes it a promising platform for the development of novel diagnostic and analytical tools.

Acknowledgements:

This work was partially supported by the National Council for Scientific and Technological Development--CNPq (n. 309240/2022-0), Coordination for the Improvement of Higher Education Personnel--CAPES (n. 88887.711903/2022-01), Research Foundation of Alagoas--FAPEAL (n. APQ2022021000004).

Ag-sensitized Eu^{3+}-doped luminescent zeolites forsensing

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Zeolites are crystalline porous materials with significant applications in greenhouse gas capture like $CO_2$and NO_x, heavy metals, hydrocarbons and other dangerous contaminants for the environment. Moreover, the porous crystalline nature of zeolites allows a homogeneous dispersion of luminescent species, which could add sensing functionality to the material. Rare earth ions (RE^{3+}) are particularly interesting for this scope because their photoluminescence (PL) emissions have a clear and identifiable spectral shape and long luminescence lifetimes. Among them, Eu^{3+}emission in the red is sensitive to the surrounding environment, potentially responding to the absorption or desorption of contaminant species. In this work, two different commercial zeolites (5A and 13X) were doped by Eu^{3+}ions and Ag aggregates via ion-exchange in solution. Ag can provide a significant increase of the Eu^{3+}PL intensity and a widening of the excitation band, as well as an additional blue emission. Optimization of the synthesis process supported by a detailed structural and optical characterization as well as preliminary testing on the sensing capabilities will be presented.

Carbon Dots from Cysteine for pH sensing, CMCdetection and cellular imaging applications.

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Carbon quantum dots (CDs) have emerged as promising nanomaterials in several scientific fields due to their small, low cytotoxicity, luminescent properties, ease of synthesis, and water solubility. Their fluorescence property is the most significant and contributed to their discovery. In this work a simple synthesis was presented, through the hydrothermal synthesis process, using cysteine as a precursor material, which results in CDs self-doped with oxygen, nitrogen, and sulfur (cys-CDs), adapted from Zhang et al. [1]. Subsequently, sensitivity was characterized and evaluated against different pHs, showing an efficient sensitivity between pHs 2 to 9, with application in real samples, and promoting control of its luminescent properties. This variation was characterized, showing a change not only in the intensity but also in the emission wavelength of the luminescence. Using various techniques, we were able to understand the causes of this effect. Rapid test devices were successfully developed for water pH analysis, as well as for the production of ink anticounterfeiting CDs [2]. The CDs derived from cysteine also were employed as a potential probe for identifying critical micellar concentrations (CMCs) of surfactants. This approach demonstrated a simple and effective technique for CMC detection. In addition, our research group conducted an investigation into the impact of solid-state thermic treatment (STT) at temperatures ranging from 303 K to 453 K on the, surface characteristics, fluorescence properties, and cellular cytotoxicity of cys-CDs. The findings of our study highlight that a simple post-synthesis treatment can effectively modify the properties of cys-CDs, making them suitable for cellular applications [3]. [1] Zhang, Y. and He, J. Phys. Chem. Chem. Phys. 17, 20154-9 (2015);
[2] Work accepted to the Nanotechnology Journal (2023);
[3] Sonsin, A. et al. Nanotechnology 33, 235708 (2022).

Computational and (bio)electrochemical Study to Measure Glucose – *Ganoderma applanatum* Lectin Interactions at Sub-nanomolar Levels

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Fungal lectins are biomaterials with great biotechnological potential, but limited knowledge about their biochemical and biophysical features prevents their proper use [1]. In this work, we evaluated the *Ganoderma applanatum* lectin (GAL) as a glucose biorecognition element at sub-nanomolar levels, using artificial intelligence and electrochemical data from a homologous agglutinin to build the working reaction interface. Materials with glucose affinity are very important to identify and manage this carbohydrate in matrices where its content is critical. Biochemical tests indicated that GAL has moderate resistance to pH (4-8) and temperature (20-60 °C) changes, with its hemagglutinating activity (376.5 HU mg -1 GAL at 20 °C) better conserved at pH ≈ 7.0 / 20 °C. Seeking to offer biocompatibility and favorable electrostatic conditions, GAL was immobilized on Prussian blue-modified glassy carbon electrode, after thermal activation of the metal-complex film. The biosensor developed, henceforth called GAL/ta-PB/GCE, showed a sigmoidal response to increasing glucose concentration, which was well-fitted by the Hill equation, allowing detect it through highly stable and precise electrochemical responses (detection limit = 10.2 pmol L^{-1}; sensitivity = 0.012 $\mu A\ \mu M^{-1} cm^{-2}$). As proof of the platform's usefulness to analyze glucose in complex samples, GAL/ta-PB/GCE was successful tested to quantify it in pharmaceutical formulations, reiterating that the association between different (bio)materials, as well as the combination between theoretical and experimental data, drives important improvements in bioelectrochemical investigations performed at molecular levels.

Acknowledgments: CNPq, CAPES, Funcap and UFCA.

Reference: [1] Abrantes-Coutinho, V.E.; Santos, A.O.; Moura, R.B.; Pereira-Junior, F.N.; Mascaro, L.H.; Morais, S.; Oliveira, T.M.B.F. Colloids Surf. B Biointerfaces, 208, 112148 (2021).

Electrochemical Biosensor for the Detection of Protein–Protein Interaction

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A novel label-free electrochemical biosensor based on the zeolitic imidazole framework (ZIF-8) was developed for monitoring protein–protein interactions (PPIs) [1]. The proteinsof interest were ADAM17 and thioredoxin-1 (Trx-1). ADAM17 is considered one of the main proteases responsible for the ectodomain shedding of surface proteins [2] and is mainly associated with pathological processes of diseases [3]. Trx-1 was previously identified by mass spectrometry as an interaction partner of the ADAM17 cytoplasmic domain (ADAM17cyto), which plays a crucial role in the regulation of ADAM17 activity [4]. ZIF-8 was employed as a transducing material and simultaneously carried Trx-1 protein, followed by the deposition of increased concentrations of ADAM17cyto. Structural and morphological characterizations were used to validate and verify the formation of ZIF-8. The interaction between Trx-1 and ADAM17cyto proteins was analyzed through electrochemical impedance spectroscopy (EIS). The results indicate a linear and inverse relationship between the impedance responses at 0.1 Hz for ADAM17cyto concentrations from 0.05 to 8 µM. The proposed biosensor also displayed a significant selectivity and stability verified by using ADAM17cyto mutant and BSA as controls. As a proof-of-concept, we compared the results with a widely used type of PPI assay based on antibody recognition, the solid-phase binding (SPB) assay. The SPB assay was able to detect a significant binding only in ADAM17cyto concentrations above 0.5 µM. The results demonstrate that the developed biosensor was 10× more sensitive and reproducible than the conventional SPB assay, representing a novel strategy for detecting PPIs.

Acknowledgments:

FAPESP, CAPES, and CNPQ.

References:

[1] Chem. Mater. 2021, 33, 1293–1306

[2] Antioxid. Redox Signaling 2018, 29 (8), 717–734.[3]
Pathol., Res. Pract. 2019, 215 (6), 152410.

[4] J. Biol. Chem. 2012, 287 (51), 43071-43082.

Electrochemical nanoplatform for diagnosis of the schistosomiasis

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Schistosomiasis is a granulomatous infectious disease, also known as "Bilharziasis", "Xistosis", "Water Belly" and "Snail Disease" [1]. Schistosomiasis is a severely disabling disease that is closely related to poverty and chronic disease [2]. In Brazil, it is caused bythe parasite *Schistosoma mansoni*[3]. When symptomatic, it is a serious disorder in whichthe prognosis largely depends on early diagnosis and treatment. Thus, the development ofnew diagnostic methods for this disease is of fundamental importance. This work aims toprepare and characterize nanostructured films of polypyrrole (PPY) and titanium dioxide nanoparticles (NPsTiO2) deposited on a gold electrode for application as an impedimetric and voltammetric sensor for the detection of schistosomiasis. On the work surface, the electrodeposition of the polypyrrole film was carried out in 12 cycles in a potential range from -0.2 to +0.7 V with a sweep speed of 100 mV.s-1. Then, glutaraldehyde was added to activate the amino groups and, after removing the excess, they were immobilized with NPsTiO2. Finally, the *S. mansoni*probe was immobilized to obtain the PPY-NPsTiO2-Probe*S.mansoni*sensor. Those steps were carried out through electrochemical impedance, which proved the adsorption of the layers by presenting an increase in charge transfer resistance and through cyclic voltammetry, reflected on the variations of the peak currents. Thus, the surface proved to be capable of binding the DNA of *Schistosoma mansoni*.

Acknowledgements: Federal University of Pernambuco, Aggeu Magalhães Institute and FACEPE.

References:

[1] Brasil. Ministério da Saúde. Esquistossomose. Governo Federal. Brasil, (2021).

[2] McManus, D. P. et al. Schistosomiasis. Nature Reviews Disease Primers, 4(1), (2018).

[3] Silva Filho, José Damião da. Schistosoma mansoni em escolares e em trabalhadores do canteiro de obras da transposição do Rio São Francisco no município de Brejo Santo-CE, (2017).

Electrochemical Nanosensors for Quantification ofAminoacids: their Role as Diseases ‘Biomarkers

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Light-assisted NO_2 gas-sensing response of hierarchical WO_3 nanostructures at room temperature

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The development of efficient sensor devices could make air pollution monitoring more accessible and may be vital in accelerating much-needed reductions in air pollution. Gas sensors based on semiconducting metal oxides (SMOx) have drawn attention due to their performance in detecting noxious species. [1,2] The traditional gas sensors commonly work at around 200°C; this high-power consumption needed has become a barrier to their application. Thus, sensing materials working at room temperature are a promising alternative to achieve low-energy consumption. [2] The WO_3 compound has attracted attention as a light-assisted sensing material for detection of a wide range of analytes. Here, we will present an investigation of the microstructural and gas-sensing properties of hierarchical WO_3 nanostructures obtained via aerosol-assisted chemical vapor deposition (AACVD). XRD and FESEM analyses revealed the formation of a monoclinic structure, exhibiting morphology like-rods with dimensions varying between 80 and 400 nm, depending on the AACVD parameters used. Regarding the sensing performance, electrical measurements pointed out that WO_3 nanostructures were sensitive towards sub-ppm NO_2 levels (100 to 1000 ppb) operating at room temperature under continuous blue-light irradiation. Also, the samples were sensitive even under ambient moisture and had a higher response to NO_2 gas compared to the other investigated gases (CO, H_2, and NH_3). The results presented here demonstrate the potential of WO_3 nanostructures as a sensing material operating at room temperature.

Acknowledgments:

We thank the financial support of CNPq (310170/2021-4) and FAPESP (2021/07214-2)

References:

[1] È. Navarrete, et al. Sensors Actuators B Chem. 298, 126868 (2019)

[2] J.V.N. de Palma, et al. Sensors and Actuators Reports. 4, 100081 (2022).

Agaricus bisporus lectin/poly(methylene blue)-based photoelectrochemical biosensor for selective lactose analysis in foodstuffs

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Agaricus bisporus mushroom contains a fascinating lectin, here defined as ABL, which has great specificity towards lactose biorecognition [1,2]. In this work, we explored this feature to develop a photoelectrochemical biosensor that recognizes ABL-lactose interactions. Biochemical and biophysical data showed that the macrofungal extract retains lectin activity (640 HU mL^{-1}) even at critical pH values (4-10) and temperatures (20-100 ℃), allowing its direct use as an ABL source. Quantum-chemical simulations and experimental studies revealed favorable electrostatic and biocompatible conditions to immobilize ABL on poly(methylene blue)/fluorine-doped tin oxide-coated glass substrate, resulting in the ABL/PMB/FTO biosensor. In addition to improve conductivity, PMB adds photoactivity (E_{gap} = 1.65 eV) to the device, allowing it to detect lectin-carbohydrate interactions with greater sensitivity under visible light irradiation (470 nm LED). Electrochemical impedance spectroscopy data showed a sigmoidal profile well-fitted by the Hill equation, expanding the working dynamic range (15-540 nmol L^{-1}lactose; LOD = 20.2 pmol L^{-1}) and avoiding laborious sample dilution or pre-concentration steps. Under optimized photoelectrochemical conditions, the ABL/PMB/FTO biosensor showed remarkable signal stability, accuracy, specificity, and selectivity to detect lactose in commercial milk formulations. This work raises even more interest in using fungal lectins as a strategic biomaterial in technologies developed to screen carbohydrates and glycoconjugates in complex samples.

Acknowledgments:

CNPq, CAPES, Funcap, UFCA, and FCT/MCTES - Portugal.

References:

[1] Singh, R.S.; Walia, A.K.; Kennedy, J.F. Int. J. Biol. Macromol. 151, 1340-1350 (2020).

[2] Abrantes-Coutinho, V.E.; Santos, A.O.; Moura, R.B.; Pereira-Junior, F.N.; Mascaro, L.H.; Morais, S.; Oliveira, T.M.B.F. Colloids Surf. B Biointerfaces, 208, 112148 (2021).

NANOMATERIALS FUNCTIONALIZED WITH GRAPHENE DERIVATIVES FOR BIOSENSORSAPPLICATIONS

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Nanomaterials such as nanoparticles metallic, magnetic, carbon nanotubes, carbon dots, TiO2 nanotubes, graphene and its derivatives, has shown an easy functionalization with biological molecules such as DNA, enzymes, antibodies, RNA, c-DNA, viruses and bacteriaamong other biological systems generating a wide range of applications in order to developnew disease biosensing devices due to the low cost and speed to obtain results compared toconventional techniques. In this study we report a novel electrochemical portable biosensorfor viruses detection and quantification based on reduced graphene oxide films. The working electrode was built following the synthesis of lysozyme-reduced graphene oxide (rGO) films proposed by Graphene oxide is synthed using modified Hummer's method andchemically reduced using hydrazine and lysozyme[1]. Thin-films were produced by dip- coating deposition using rGO solution onto a gold substrate pre-treated with cysteine. Wereport the successfully application for RNA virus detection using cDNA functionalization onthe working electrode surface and quantification of RNA concentration with a linearly dependency of chronoamperometric current. Also, it shows selectivity against an RNA different from the one used for electrode functionalization. This novel biosensor showed tobe an innovative electrochemical method for in-situ diagnosis of rabies disease in bats witha fast response [2]. This work is grateful to the Fapesp Process : 2020/10567 andCNPq/MCTI/FNDCT 22/2022-Linha 2, Process: 408397/2022-5.

1. J. Zuñiga, et. al, Ana Champi, Diamond Related Materials, 126, 109093, 2022.
2. Ronaldo Challhua, et. al , Ana Champi, Biosensors Bioelectronics, 2023.

Nanostructured sensor based on multi-walled carbon nanotube and temporin for bacterial detection

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Nosocomial infections and antimicrobial resistance remain recurring problems. Rapid identification of microorganisms is essential to direct immediate treatment and epidemiological control. Electrochemical biosensors identify pathogens and clinically significant biomarkers as an innovative alternative combining nanotechnology with various biorecognition elements such as antimicrobial peptides. The present work aimed to develop an impedimetric biosensor based on multilayer carbon nanotubes and the antimicrobial peptide Temporina-PTA to detect bacteria of clinical interest. Through electrodeposition, the MWCNTs coated the electrode surface with gold. Subsequently, T-PTA was immobilized through coupling agents. The electrochemical impedance spectroscopy technique evaluated the interaction of the sensor platform with bacterial suspensions. Gram-negative and gram-positive bacteria were evaluated. The biosensor presented a detection limit of 10 CFU/mL for all studied species. The analysis time is 5 minutes. Gram-negative species, especially E. coli, obtained the highest impedimetric response. Electrochemical biosensors based on antimicrobial peptides show promise in identifying bacteria, getting results in a few minutes and demonstrating remarkable sensitivity. The developed detection strategy has great potential to help establish technologies to detect pathogens of clinical interest, such as enzymatic and molecular assays.

Acknowledgements: Universidade Federal de Pernambuco e CNPq.

References:

[1] A.G. da Silva-Junio, I.A.M. Frias, R.G. Lima-Neto, L. Migliolo, P.S. e Silva, M.D.L. Oliveira, C.A.S. Andrade, Electrochemical biosensor based on Temporin-PTA peptide for detection of microorganisms, J. Pharm. Biomed. Anal. 216 (2022).

[2]G. Bolat, Investigation of poly(CTAB-MWCNTs) composite based electrochemical DNA biosensor and interaction study with anticancer drug Irinotecan, Microchem. J. 159 (2020) 105426.

Photochromic Sensing of La^{3+} and Lu^{3+} Ions using Polycaprolactone Fibers Doped with Spiropyran Dyes

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There is a constant risk of contaminated water with toxic pollutants like heavy metal ions, anions, organic compounds, dyes, lanthanides, gases from anthropogenic and natural activities. Even small amounts of toxic compounds can pose a risk to the environment and human health because they are not biodegradable and bioacumulate. As a result, it is critical to monitor heavy metals in water such as lanthanide ions, but traditional analytical methods are limited due to the cost and needs of large equipment. To address this challenge, it is important to develop an economical, real-time, accurate, and selective detection of toxic pollutants in natural environments. Here, we report three spiropyran derivatives, which were then incorporated into PCL electrospun fibers as a platform for sensing heavy metals in water. The materials obtained were then screened for all lanthanide ions, and the three polymeric systems showed distinct colorimetric responses to different ions, especially Lanthanum and Lutetium. We also demonstrated that it is also possible to selectively identify and differentiate $La^{3+}$and $Lu^{3+}$by emission and naked eye detection. These results showed a promising prospect for portable sensor for field use that may act as colorimetric sensors of toxic heavy metals. Overall, this study demonstrates the potential of spiropyran derivatives as versatile and tunable materials for surface modifications with various applications. Acknowledgements:
This work was supported by Brazilian agencies CNPq and FAPEMIG, Biosmart Nanotechnology, Doaplex, LAREMAR-UFMG, Center of Microscopy-UFMG and GEM-UFU.

References:
[1] R. Klajn. Chemical Society Reviews, 43, 148-184 (2014)
[2] R. C. L. Machado, F. Alexis, F. B. de Sousa, 24, 4243 (2019)
[3] G. C. Silva, A. C. Bertoli, H. A. Duarte, A. C. Q. Ladeira, 10, 108715 (2022)

Photopolymerizable photonic nanocomposites for personal dosimeters to monitor cutaneous productionof vitamin D

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This work presents a photopolymerizable printable nanocomposite consisting of a polymeric structural passive phase and a europium complex that acts as a photonically active phase: Eu(btfa)$_3$·bipy@resin. This nanocomposite is being used to produce a printable photonic device that accumulates and reports the dose of UV radiation received by a user, formonitoring their sun exposure as a function of their skin type [1]. Cutaneous production ofvitamin D3 can provide more than 90% of the daily dose, and as it is catalyzed by solar UVBradiation, to monitor the dose received by the user and correlate it with their vitamin D3production, a printed molecular dosimeter was developed here to produce a photonic signalas input for conversion calculations in a software designed to run on a smartphone, [2] forpersonal monitoring of vitamin D3 production. This device was printed in the form of smalldisks using a mask stereolithography 3D printer (MSLA) and the active nanocomposite useda commercial resin with Eu(btfa)$_3$-bipy, exhibiting a well-behaved luminescence quenching related to the absolute UVB dose exposure [3]. The conversions are performed in a smartphone that reads the Eu^{3+}transition peaked at 612 nm ($^5D_0 \rightarrow {}^7F_2$), under a short UVA LED pulse. The thickness of the printed device can be determined as a function of sensitivityand the time of its useful life required for use, resulting in a parameter to be considered inthe dose conversion, together with other parameters associated with the user, such as skintype, age, exposed fraction of the skin, informed in the personal monitoring App.

Acknowledgments:
This work was supported by SibratecNano - FINEP/MCTI, CNPq and CAPES Ed. 9/2020.

References:
[1] P.A. Santa Cruz. BR, n. PI 1003026-3, Letters Patent issued 09/21/2021
[2] E.C.R. Vaz et al., Braz. J. Radiat. Sci. 10-02A, 01-16 (2022)
[3] F.L.N. Souza et al., Mater. Res. Express 3(4), 045701 (2016)

Physical-chemical characterization of biochar from cupuassu bark (Theobroma grandiflorum) for application as a soil conditioner

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Biochar is the product of pyrolysis of renewable biomass, with low oxygen availability. This material is very porous, rich in carbon and contains a large amount of organic structures and inorganic elements. The chemical composition of the ash fraction consists of macro and micronutrients for plants and improves soil fertility conditions. The objective of this research was to carry out the physical-chemical characterization of the cupuaçu bark biochar, identifying its physical properties favorable to its use as a soil conditioner. The cupuaçu peels (Theobroma grandiflorum) used as raw material was purchased from farmersand fairgrounds in the municipality of Parintins. The synthesis of biochar for physical- chemical and morphological characterization was carried out using the activating agent phosphoric acid (H3PO4) in a ratio of 1:1 by mass. The physical and chemical characterization by X-Ray Diffraction Technique (DRX) was performed at Federal Institute of Amazonas, Campus Manaus Centro and the morphological one by Scanning Electron Microscopy (SEM) at INPA''s Optical Microscopy Laboratory. the X-ray diffractograms obtained for the biochar samples synthed at 400°C and 500°C. It is possible to observe thatthe samples have an amorphous structure, characterized by the wide peak in the 2θ positionfrom 5° to 30° C. The amorphous peak is attributed to the presence of carbon. The micrographs of biochar synthed at 400°C and 500°C show the morphology of the material. Itis possible to identify that the surfaces are porous. Figures 2 A and C corresponding to thebiochar synthed at 400°C show larger pores than the biochar synthed at 500°C. It can beconcluded that the lower temperature in the fast pyrolysis influences the formation of largerpores. This may indicate that the raw material of the samples was thermally decomposedduring the pyrolysis process, as expected, and that the obtained biochar has a high capacityfor nutrient and water retention.

Planar vs Non-Planar Orientation in AuAg-Catalyzed InP Nanowire Growth: Implications for Sensing Device Fabrication

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The growth of nanowires using metal catalysts presents a challenge for producing devices on a larger scale, especially when integrating III-V nanowires with Si-based electronics. The correct positioning of vertically-grown nanowires on planar electrodes is still a problem if a high yield in device fabrication is required, as we demonstrated in the past with InP biosensors [1]. To address these issues, controlling the orientation of nanowires in or out of the substrate plane using different metal catalysts may be a useful tool. In this study, we investigated InP nanowires catalyzed by Au, Ag, and AuxAg1-x nanoparticles grown on GaAs substrates with varying orientations. Our analysis of experimental data and modeling revealed that the droplet surface density-to-In precursor flux ratio is a crucial parameter in controlling the nanowire growth mode and spatial orientation. These findings offer insight into the vapor-liquid-solid growth of planar nanowires and provide a means of controlling the growth orientation of III-V nanowires catalyzed by different metallic nanoparticles on substrates with lattice mismatch.

Acknowledgments:

This research was supported by the Brazilian funding agencies CNPq and Fapesp (grant number 2019/07616-3).

References:

[1] R. Janissen et al, Nano Letters 17, 5938-5949 (2017)

Red Wine and Drinking Water Analysis by A-TEEM Molecular Fingerprinting Spectrocopy: Free of Consumables Classification of Phenolics and Analysisof Colored Dissolved Organic Matter Compounds thatCause Toxic Disinfection By-Products.

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A-TEEM Spectroscopy is a unique technology for quality analysis of drinking water, wine and may other agricultural inputs and products. A-TEEM Spectroscopy simultaneously measures the absorbance spectrum and fluorescence (EEM) of Colored Dissolved Organic Matter (CDOM) compounds. A-TEEM Spectroscopy reports NIST-traceable fingerprints which can be evaluated using multivariate statistics such as PARAFAC (Parallel Factor Analysis), PCA (Principal Components Analysis), CLS (Classical least Squares) and PLS (Partial least Squares Analysis). For wine characterization, importantly A-TEEM fingerprints yield qualitative and quantitative composition of key flavor and color determinants in wine and spirits that are not discernible with simple Absorbance or Transmission data analysis. Of the hundreds of different compounds that have been identified in grapes, it is the phenolic content of ripening grape berries that fundamentally determines the quality of a wine.The different classes of phenolics (anthocyanins, tannins, flavonols, catechins) affect the color, the mouthfeel, flavor and aroma to various extent. The individual compounds comprising these classes of phenolics contribute in concert to give the wines their uniquecharacter. The simultaneously acquired Absorbance,Transmission and IFE-corrected EEMdata can be used to evaluate lot-to-lot, regional, and varietal characteristics. For water analysis, Colored Dissolved Organic Matter (CDOM) compounds plays an important role inEPA regulations for drinking water sterilization. The concentration of CDOM compounds inthe humic and fulvic acid classes are regulated because they generate toxic DBPs during halogen disinfection treatments for microorganisms such as Giardia spp. Key parameters of interest includes the absorbance at 254 nm (A254 nm) used in the Specific UV absorbance calculation to predict DBPs and the humic/fulvic/protein like peaks (A/C/T) also correlated with DBPs certified by either NIST and or ISO traceable.

Scalable and green formation of graphitic nanolayers produces highly conductive pyrolyzed paper toward sensitive electrochemical sensors

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While pyrolyzed paper (PP) is a green and abundant material that can provide functionalized electrodes with wide detection windows for a plethora of targets, it poses long-standing challenges against sensing assays such as poor electrical conductivity, with resistivities generally higher than 200.0 mΩ cm (e.g., gold and silver ~0.2 mΩ cm). In this regard, the fundamental hypothesis that drives this work is whether a scalable, cost-effective, and eco-friendly strategy is capable of reducing the resistivity of PP electrodes toward the development of sensitive electrochemical sensors, whether faradaic or capacitive. We address this hypothesis by simply annealing PP under an isopropanol atmosphere for 1 h, reaching resistivities as low as 7 mΩ cm. The annealing of PP at 800 or 1000 °C under isopropanol vapor leads to the formation of a highly graphitic nanolayer (~15 nm) on the PP surface, boosting conductivity as the delocalization of π electrons stemming from carbon sp2 is favored. The reduction of carbonyl groups and the deposition of dehydrated isopropanol during the annealing process are hypothesized herein as the dominant PP graphitization mechanisms. Electrochemical analyses demonstrated the capability of the annealed PP to increase the charge-transfer kinetics, with the optimum heterogeneous standard rate constant being roughly 3.6×10^{-3}cm s^{-1}. Freestanding fingers of the annealedPP were prototyped using a knife plotter to fabricate impedimetric on-leaf electrodes, ensured the real-time and in situ monitoring of the loss of water content from soy leaves, outperforming non-annealed electrodes in terms of reproducibility and sensitivity. Such an application is of pivotal importance for precision agriculture and development of agricultural inputs. So the new conductive PP in a scalable, low-cost, simple, and eco-friendly way, providing new opportunities to sensitive electrochemical devices.

Study of a ZnO gas sensor produced by slot-die coatingand using atmospheric pressure plasma treatment

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Recently, interest in flexible and printed electronic devices has grown due to their application advantages, such as wearable technologies that can be integrated into the Internet of Things (IoTs) [1]. It is interesting to integrate the sensing of gases, arising from volatile organic compounds, as wearable devices, which can be attached to badges, for example. In this context, atmospheric pressure plasma (APP) technologies are of interest asa way of deposition, treatment, or surface modification of flexible materials, as vacuum equipment is not necessary, with low process cost [2]. With this objective, to integrate the use of sensors in flexible devices, the gas sensor based on ZnO operating at room temperature and the study of the modifications created by the APP in the treatment of the substrate and device will be presented [3]. Optical, morphological, and electrical characterizations of the film used in the active layer of the sensor and of the substrate and device treated by plasma were carried out. For the performance of the devices, measurements of sensitivity, response as a function of temperature, and selectivity of alcohol vapors were carried out.

Acknowledgment: This work has been supported by CAPES and CNPq.

[1] Park, J., et al., Wearable, wireless gas sensors using highly stretchable and transparent structures of nanowires and graphene. Nanoscale, v. 8 (20), p. 10591-10597, (2016).
[2] Kim, K. N.; Lee, S. M.; Mishra, A. ; Yeom, G. Y. Atmospheric pressure plasmas for surface modification of flexible and printed electronic devices: A review. Thin Solid Films, v. 598, p. 315-334(2016).
[3] Zhu, L. and Zeng, W. Room-temperature gas sensing of ZnO-based gas sensor: A review. Sensors Actuators A Phys., v. 267, p. 242-261 (2017).

Synthesis and characterization of electrocataliticsurface for application in biosensors

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The present describes an electrocatalytic film consisting of a self- assembled monolayer functionalized with ferrocene (Fc). The synthesis was carried out in a gold electrode, from the chemisorption of 2-aminoethanethiol (CIS), followed by o-Phenylenediamine (OPD) binding via the presence of a dicarboxylic acid. The Fc was carried out to the film via OPD through π-π interactions which resulted in a stable functionalization [1]. The electrocatalytic activity of the Fc film was confirmed at the electrode-electrolyte interface by a cyclic voltammetry technique, in the presence of support solution (0.1 M KCl). Electro-oxidation is observed at a redox potential of 0.34 V and 0.15 V for the oxidation and reduction peaks, respectively [2]. As a proof of concept, the electrocatalytic film was mounted over the configuration of a field effect transistor (FET), and it was possible to detect the presence of bovine serum albumin by the change in the drain current measured. The ferrocene-functionalized film stages were all electrochemically characterized by cyclic voltammetry technique. The electrocatalytic film developed shows up as a potential for application in immunosensors, since the electrooxidation potentials do not compromise the activity of biomolecules. In addition, the use of the film has the advantage of dispensing redox probes or the use of marked antibodies in voltametric readings [3].

Synthesis and characterization of Eu-doped ceria nanostructures with controlled morphologies for humidity sensing

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Over the last decade, particular interest has been devoted to the morphology-controlled synthesis of various metal oxide semiconductors to investigate the correlation between surface modifications and properties [1]. In this work, Eu-doped CeO2 nanostructures with sphere, rod, cube, and polyhedron morphologies were synthesized via the microwave-assisted hydrothermal (MAH) method. The samples were characterized by multiple structural and optical techniques. XRD analysis showed that ceria's fluorite-type structure was formed for all samples, without secondary phases. Raman spectroscopy analysis showedthat oxygen vacancies were the main defect in the samples. SEM and TEM microscopiesshowed the morphologies evolved from spherical nanoparticles to nanocubes by increasingthe synthesis temperature. These nanostructures were then used to fabricate films by the screen-printing technique. The nanostructures were mixed with organic vehicles to optimizethe deposition process and enhance the adhesion to the substrate. The films were depositedonto alumina substrates with interdigitated gold electrodes and heat-treated to eliminatethe organic compounds. The films were characterized by SEM and had their electrical properties measured under different temperature and relative humidity (RH%) conditions. The films were porous and without cracks or other discontinuities. Increasing the temperature enhanced the response of the films to humidity. The films were able to detect different RH% and the nanorod-based film displayed the best performance for humidity sensing compared with the other samples.

Acknowledgements: The authors thank the Brazilian agency FAPESP (2018/26550-0) for their financial support to this project.

References:

[1] M. Hamanaka, K. Imakawa, M. Yoshida, Z. Zhao, S. Yin, X. Wu, Y. Huang, J. Wu, T. Sato, Synthesis and gas sensing properties of SnO2 nanoparticles with different morphologies, J. Porous Mater. 23 (2016) 1189-1196.

Synthesis and Characterization of Silver Nanoflowers and Silver Nanostars and their Application as SERS substrate for Pesticides Sensors in Aqueous Solutions

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Metallic nanoparticles such as silver nanoparticles (AgNP) have been of great interest in recent years due to their highly interdisciplinary applicability, since they have very characteristic physical and chemical properties such as strong light absorption and, because of their capacity of producing surface plasmon resonance (SPR) effect through excitation of electron oscillation vibrational modes, which yields intensification of Raman spectra (SERS - Surface-enhanced Raman Scattering - effect), makes them great materials for sensing the presence of organic molecules, such as pesticides, in aqueous solutions even though they present very low concentration limits in the order of ng/L [1]. Because of that it is of extreme importance to obtain well synthed substrates capable of supporting these nanoparticles and making possible their application and further detection and identification of pollutants in water resources [2]. Therefore, the present work proposes the engineering of thin films composed of different morphologies (star and flower) silver nanoparticles through the deposition of this materials onto glass substrate using blade coating method to detect the presence of atrazine, thiamethoxam and imidacloprid pesticides in aqueous solutions. UV-VIS spectra, dynamic light scattering (DLS) measurements, atomic force microscopy (AFM), and scanning electron microscopy (SEM) confirmed the morphologies and topography of AgNP films. Due to plasmon effect of AgNP the pollutant models were detected at very low concentrations (10^{-4}~10^{-6}M) in an aqueous solution by SERS effect. These promising results augur well for further developments of AgNP plasmonic thin films for organic pollutants detection.

Acknowledgments:
CAPES and FAPESP supported this work.

[1] A. Amoriani; F. Firouzi; D. F. Haghshenas. Plasmonics, 15, 1077-1082 (2020).
[2] R. de Oliveira; W. da S. Martini; A. C. Sant'ana. Environmental Nanotechnology, Monitoring & Management, 17, 100657 (2022).

The effect of gold nanoparticle electrodeposition time in electrochemical immunosensing: a study based on S-protein biosensors

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It is known that the use of nanoparticles in (bio)sensing can significantly improve analytical performance through catalytic properties, or the increase in the number of bioreceptors in the electrode surface, for example. In this work, we have explored the influence of gold nanoparticles (AuNPs) electrodeposition time on the analytical parameters of electrochemical biosensors. For that, AuNPs were added to screen-printed carbon electrodes (SPCEs) using electrodeposition times ranging from 9 to 90s, providing AuNPs with different diameters (143 ± 31 to 752 ± 207). The platforms were modified with anti-S protein antibodies, allowing the label-free detection of S protein from both SARS-CoV and SARS-CoV-2 through electrochemical impedance spectroscopy (EIS). As expected, increased electrodeposition time led to higher concentrations of electroactive species, AuNPs with higher diameter, as well as lower charge transfer resistance. However, the the electrodeposition time was inversely correlated to the enhancement in sensitivity, specificity, and limit of detection. This is probably because thin films present lower capacitive currents, increasing the sensitivity of the devices. The biosensor built with the 9s deposition time was the one that displayed the best performance among the ones tested (9, 30 and 60s), being able to detect S protein in the range from $10^{-11}$to 10^{-7}mol/L and reaching a limit of detection of 3.16 pmol/L. The devices were used for the determination of SARS-CoV-2 inactivated viruses in real saliva samples and were able to differentiate samples from diseased individuals (10^{6}PFU/mL of SARS-CoV-2) from healthy ones. The analysis was completed in 35 min using small sample volumes (10 µL) and is estimated to cost less than US$ 0.03 per device. We expect these studies to aid in the development of future nanomaterials-based biosensors, as a means of increasing its sensitivity, specificity, and other analytical parameters. Acknowledgement Fapesp 2018/19750-3.

Titanium Oxide Nanoparticles: Synthesis andApplication in Isopropyl Alcohol Detection

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Production of oxide based chemical sensors have been widely studied, although most of them only perform at high temperatures [1]. In this study, an isopropyl alcohol sensor, working at room temperature, based in titanium oxide nanoparticles (TiO_2NPs) is proposed. The TiO_2NPs were synthetized by the Sol-Gel method, where the precursor solution was a mixture of titanium isopropoxide with isopropyl alcohol. After stirring, the precursor solution was slowly mixed with a previously prepared and heated solution of hydrochloric acid diluted in ultrapure deionized water. After cooling, the liquid was centrifuged to separate the resulting in TiO_2NPs that were later dispersed in isopropyl alcohol, as reported in [2]. The material was used as active layer in a chemical sensor for isopropyl alcohol detection by casting the TiO_2NPs dispersion on electroless nickel immersion gold (ENIG) interdigitated electrodes on glass-reinforced epoxy laminate material (FR-4) substrates. The sensor characterization was done through impedance spectroscopy measurements at different analyte concentrations in a sealed static chamber filled with nitrogen at room temperature. The best operating frequency obtained was 3 kHz, chosen considering signal to noise ratios and sensitivities in the range 100 Hz and 20 MHz. The sensitivity at 3 kHz was $5{,}93\text{x}10^{-3}\text{ppm}^{-1}$. These results indicate a possible application of titanium oxide nanoparticles as active material in volatile organic compounds sensors at room temperature.

Acknowledgements:
CAPES and PIPE-UFPR.

References:

[1] J. Bai and B. Zhou. Chemical reviews, 114(19), p. 10131-10176 (2014).

[2] A. A. B. Cárdenas and J. P. M. Serbena. CIÊNCIA E ENGENHARIA DE MATERIAIS: CONCEITOS, FUNDAMENTOS E APLICAÇÃO, vol. 1(1), p. 382-391 (2021).

$Y_7O_6F_9$ FOR CONTACTELESS TEMPERATURE SENSOR: THE RELATION BETWEEN STRUCTURAL AND OPTICAL PROPERTIES

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Contactless luminescent thermometers provide accurate and real-time temperature detection, bringing new technologies to human health diagnoses. For optical temperature detection in vivo, neodymium-doped materials are promising candidates due to their excitation and emission in biological windows, improving penetration depth in the bio-tissue. However, the relative sensitivity depends on the matrix in which the Nd is inserted, excitation wavelength, as well as on the synthesis method. For this work, two yttrium fluoride-based materials doped with 1 mol% of Nd^{3+}were produced using microwave-assisted hydrothermal (MAH) reaction. During nucleation process and posterior thermal treatment, fluoride compounds tend to absorb oxygen ions, changing their structure and, consequently, optical characteristics. Based on these changes, two crystalline matrices were obtained: $YF_3$and $Y_70_6F_9$(inspected via X-ray diffraction - XRD), with rod-like morphology for both compounds, as determined by SEM images. The oxygen content was thoroughly investigated by XPS analyses and correlated to the thermal sensitivity of the two materials. Based on LIR (Luminescence Intensity Ratio) methodology, the highest sensitivity value was achieved for 660 nm excited Nd-doped Y706F9 sample, presenting relative sensitivity of 1.6%K-1. These results indicate that materials are promising contactless temperature sensors for in vivo applications, and its direct connections with synthesis parameters.

Acknowledgments: The authors thank FAPITEC and CAPES for the scholarship grant. CMNano-UFS and LNNano for the facilities.

3D printed electrochemical sensor based on carbonblack for determination of Carbendazim in honey

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Electrochemical sensors obtained by additive manufacturing have gained prominence in the scientific community due to the possibility of large-scale production, the use of sustainable/renewable materials, and the relatively low cost [1]. In addition, in recent years, together with additive manufacturing, the production of conductive filaments has gained more and more space, allowing the manufacture of custom-made sensors with the most varied composition [1,2]. Such 3D printed sensors have shown remarkable performance and applicability in the most different areas of society, such as the detection of pesticides in food matrices. In this regard, the present work applied a lab-made conductive filament based on carbon black and polylactic acid to produce an electrochemical device for detecting carbendazim (CBZ) in honey. The manufactured electrochemical sensor was electrochemically treated in a basic medium to remove excess PLA of the surface and expose the conductive material. Thus, the sensor showed a superior response for CBZ detection. Thus, an analytical curve was built in the range from 0.5 to 20.0 μmol L-1 with a limit of detection of 0.1 μmol L-1. Furthermore, recovery tests carried out in honey samples from fortification with three known concentrations showed values close to 100%, demonstrating that there is no significant matrix effect. Therefore, it is possible to infer that the production of electrochemical sensors from lab-made conductive filaments was successfully reached, demonstrating the applicability of the material since it was possible to determine CBZ easily and simply in sub-micromolar concentration.

Acknowledgments:FAPESP (2017/21097-3 and 2022/06145-0); CAPES (financial code 001; 09/2020 and88887.504861/2020-00); CNPq (301796/2022-0 and 380632/2023-3)

References:[1] Stefano, J.S., et al., Anal. Chem. V. 94, pp. 6417-6429, 2022.[2] Stefano, J.S., et al., Anal. Chim. Acta., 1191, e339372, 2022.

Additive manufacturing sensor by dual extrusion for fenitrothion detection

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Electrochemical sensors built by additive manufacturing have been applied in several research areas. Researchers have exploited the ease of modeling, rapid prototyping, and high definition offered by this technique, together with conductive filaments, to produce new sensor architectures.[1] Dual extrusion has grown in this segment, offering the possibility of working with two materials simultaneously, avoiding defects caused during the assembly of a device produced separately. In this work, a device built by dual extrusion additive manufacturing, using thermoplastic and conductive filaments based on carbon nanomaterial, was proposed for the detection of the pesticide fenitrothion in honey samples for monitoring its application in the field and as quality control in food samples.[2] The construction of the device was carried out using a 3D printer with the Fused Deposition Modeling technique, which was subsequently treated with dimethylformamide and washing it in ethyl alcohol and an ultrasonic bath in ultrapure water.[3] The morphological characterization of the sensor was performed before and after the treatment by scanning electron microscopy, which is possible to visualize the removal of Polylactic Acid from the surface and exposure of carbon black. The square wave voltammetry technique was used, in which the sensor reached the highest peak current for fenitrothion detection. Under optimized parameters, the analytical curve was built, comprising a linear concentration range from 4.0 to 200 µmol L^{-1}, and a limit of detection of 1.3 µmol L^{-1}. The proposed sensor is being applied to honey samples, proving to be effective in detecting fenitrothion.

Acknowledgments:
FAPESP (2017/21097-3 e 2021/03179-8), CAPES (001, 09/2020, and 88887.504861/2020-00) and CNPq (301796/2022-0).

References:

[1] S.W. Kwok, et al., Appl. Mater. Today, 9, 167-175 (2017)
[2] L.J. Unnevehr, et al., Food Policy, 22, 105-106 (1997)
[3] J.S. Stefano, et al., anal. chem., 94, 6417-6429 (2022)

A fluorescent biosensor based on hydroxyapatite nanoparticles for tuberculosis biomarker detection

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Tuberculosis (TB) cases and deaths have increased in Brazil since last year [1]. Early detection is crucial for the effectiveness of treatment. The standard method for diagnosis, the mycobacterial culture, has a slow growth rate (about 8 weeks), which can worsen the patient's health condition [2]. An alternative for fast and sensitive diagnosis is using biosensors based on nanomaterials due to their unique properties. Hydroxyapatite nanoparticles (HANps) have easy adsorption of molecules on their surface and exhibit photoluminescence (PL) by manipulating defects in their structure [3]. This project aims to develop a fluorescent biosensor for the detection of the CFP10/ESAT6 complex, a TB biomarker. HANps were synthed following the method established by Machado et al [3] and functionalized with polyethyleneimine (PEI). Abs against CFP10/ESAT6 were covalently bonded to HANps-PEI and BSA was used as a blocking agent. Fourier-transform infrared spectroscopy, surface zeta potential, and PL spectroscopy characterized each functionalization step, indicating the successful Abs immobilization onto HANps. Significant changes in the intensity of the PL spectrum step were noticed. The results have shown that the nanosensor is promising to be used as a detection probe for the accurate, fast, and low-cost diagnosis of TB.

Acknowledgments:

The authors acknowledge the financial support provided by the National Council for Scientific and Technological Development - CNPq (Grant number 440116/2020-1, 382396/2022-7) and the cooperation of the researchers from GNano in our studies.

References:

[1] B. Epidemiológico, Tuberculose | 2022.

[2] Tan, Y. et al. An automated smear microscopy system to diagnose tuberculosis in a high-burden setting. Clinical Microbiology and Infection 25, 1553-1559, 2019.

[3] Machado, T. R. et al. A novel approach to obtain highly intense self-activated photoluminescence emissions in hydroxyapatite nanoparticles. J Solid State Chem 249, 64-69, 2017.

AMPEROMETRIC DNA BIOSENSOR FOR DETECTION OF CHILDHOOD LEUKEMIA WITH POOR PROGNOSIS MUTATION

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Pediatric acute lymphoblastic leukemia (ALL) is a hematopoietic neoplasm responsible for high mortality in children. The current diagnosis is based on a series of investigations, however, for clinical decision-making, genetic markers are decisive. Despite this, these genetic methods need a long time to respond and more than one is needed to confirm the neoplasm. The translocation between chromosomes 4 and 11 (t(4;11)) is a poor prognosis mutation in childhood [1]. Biosensors are tools that provide fast response with easy instrumentation, especially when associated with electrochemical transduction. In addition, materials such as conductive polymers and nanoparticles are introduced in the structuring of biosensors to improve bioanalytical performance [2]. In this context, our objective is the development of a DNA biosensor to detect t(4;11) in pediatric patients with ALL. For this, the electropolymerization of PPy was carried out on a surface of flexible working electrode. As a second step, the quantum dots were adsorbed followed by primer immobilization to t(4;11). Biorecognition assays were performed with plasmid samples. Cyclic voltammetry was used to characterize the steps of construction and evaluation tests. As a result, voltammetric changes were observed for each self-assembly and biorecognition step. With the ΔI% it was possible to observe the hybridization process. Thus, the DNA Biosensor developed proved to be suitable for use in the diagnosis of ALL.

Acknowledgments:

FACEPE, CNPq and CAPES.

References:

[1] Madhusoodhan, P. P., Carroll, W. L., Bhatla, T. (2016). Progress and prospects in pediatric leukemia. Current problems in pediatric and adolescent health care, 46(7), 229-241.

[2] Zhu, Q., Liu, J., Wang, X., Huang, Y., Ren, Y., Song, W., ... Liu, C. (2020). Polypyrrole functionalized graphene oxide accelerated zinc phosphate coating under low-temperature. ES Materials Manufacturing, 9(6), 48-54.

Assessment of ZnO and ZnO-rGO-ZnO sensor'sproperties for ozone detection

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The development of nanotechnologies and new 2D materials has enabled a search for faster, more selective, and portable gas sensors that are capable of meeting the demands of areas such as industrial safety and environmental monitoring. In this context, due to its high electronic mobility and large surface area, graphene-based materials show promise for use as gas sensors [1]. Currently, several strategies are developed to improve the sensing properties of ZnO, one of these techniques is the combination of ZnO with reduced graphene oxide (rGO). Thus, this research aims to prepare sensors from ZnO and its combination with rGO, to evaluate the ability of these samples to detect ozone. ZnO was synthesized by controlled precipitation, using ammonium hydroxide as a precipitating agent and zinc acetate as a precursor [2]. The sensors were manufactured by drop casting ZnO solutions and rGO dispersion on interdigitated platinum electrodes, being named ZnO and ZnO-rGO-ZnO sensors. By XRD, the formation of a compact hexagonal crystalline structure of the ZnO wurtzite type was verified without the presence of impurities. ZnO SEM images showed that needle morphology was produced. Measurements of gas detection at different temperatures demonstrate that both sensors present a higher response at 200 °C and that the ZnO-rGO-ZnO sensor presented an average response 30 times greater than that of the ZnO sensor.

Acknowledgments: This research was funded by Sao Paulo Research Foundation's financial support - FAPESP (grants N° 2022/06404-5, 2019/22076-5, 2022/03325-7, 2023/00913-8, and 2013/07296-2). The authors are also grateful to the Brazilian National Science Financial Agencies CNPq and Capes.

References:

[1] F. Schedin et al. Detection of individual gas molecules adsorbed on graphene. Nature Materials, 6, 652 - 655, 2007.

[2] M. Montero Munoz et al. Growth and formation mechanism of shape-selective preparation of ZnO structures. Physical Chemistry Chemical Physics, 22, p. 7329, 2020.

Autonomous irrigation system powered by organic solar panel (OPV)

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Faced with the need to expand food production, due to population growth, and global warming with more irregular rainfall, the relevance of irrigation in agriculture has increased. In this sense, new technologies to increase the efficiency of water use with lower costs are needed. Our project aims to develop an automation system, powered by solar energy, with automatic control function (on and off) of the water flow in an irrigation system for small rural properties. We acquired equipment to set up the automation system with a low-cost arduino microcontroller, model uno, powered by an off-grid solar system [1] and connected to sensors responsible for measuring soil and air humidity, which will be used in the activation autonomous from the irrigation pump. First, we use an inorganic silicon solar panel. Later, we will use an organic solar panel (OPV) based on carbon: lighter, more flexible, semi-transparent and more sustainable than traditional models. The effectiveness of the prototypes developed in our laboratory will be carried out with data collection in a low-income family property in the southern region of Tocantins. Thus, it is expected to offer a viable alternative to reduce operating costs in an automation system.

Acknowledgements

IFTO/ IFES/ USP/ EESC/ IFSC

References

[1] Testezlaf, R. Use of irrigation in protected environments: care and attention. ITEM. Irrigation and Modern Technology, ABID, Brasília, DF, v. 53, p. 18-22, 2002.

Beeswax coating: Sustainable alternative for waterproofness in electrochemical sensing

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The COVID-19 pandemic exposes the limitation of access to accurate devices for monitoring viral diseases. Hence, searching for new monitoring devices, focusing on accuracy, simplicity, and low cost, has been exhaustively done [1]. On the other hand, the development of new, more sustainable analytical platforms become another challenge, aiming to avoid environmental problems generated by sensor discarding. This work focuses its efforts on the construction of a biodegradable and waterproof substrate for the manufacture of SPEs, using commercial inks. Here, we focused on the use of beeswax (BW) to make commercial paper waterproofed (WPpaper) for subsequent screen-printing manufacturing. The SPEs were printed in polyester, non-waterproof paper, commercial universal spray protector water-repellent, and beeswax-based waterproof paper substrates, respectively, in which the BW promoted the best waterproofness to the paper. Structural and chemical properties of all substrate and electrode surfaces were characterized by XPSand SEM. Their electrochemical performance is benchmarked against the near-ideal outersphere redox probe hexaamineruthenium (III) chloride. The WP paper exhibited a HET rateconstant 133% higher than the non-WP paper. Regarding the waterproofness, only 40 μg ofBW per electrode increased the water contact angle from 96.8° to 126.6°, considered highlyhydrophobic. Finally, after cyclic voltammetry analysis, the SPEs WPpaper-based presented43.3% less current variation, after 100 cycles, compared to the non-WPpaper, similar to results obtained for the SPEs polyester-based. Therefore, WPpaper could be used as a cheaper and environmentally friendly alternative to commonly used substrates, such as polyester.

Acknowledgements

FAPESP and Capes.

References

[1] Brazaca, L. C., et al. Analytica Chimica Acta, 1159, 338384 (2021).

Biodegradable electrode from polyvinyl acetate and carbon lignin (PVA-lk) to detect the fungicide Carbendiazin.

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The agrobusiness is one of most important economic sectors in Brazilian economy, being also one of the most polluting sectors causing environmental and social impact. Many producers dispose irregularly pesticides and mishandle them, polluting the soil and water bodies(SINGH et al., 2016). Sensors are applied to detect pesticides to point out which substance is being used in that culture and to inspect whether the producer is within the legislation(KILLEDAR et al., 2022). This work studies a sensor based on polyvinyl acetate and carbon lignin (YAO et al., 2022), being biodegradable and dismissing the use of organic solvents to detect Carbendiazin, a wide range fungicide applied in fruit and cereal crops. The carbon lignin was obtained but carbonization of lignin kraft in nitrogen atmosphere, then added to PVA to obtain a conductive film to build the sensor up. The sensor is investigated using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) and Electrochemical study (cyclic voltammetry and Electrochemical Impedance Spectroscopy). The results are analyzed by analysis of variance (ANOVA) to verify significant detection of Carbendiazin applying the proposed sensor. SEM makes it possible to observe the distribution of lignin though the polymeric matrix, EDS pointed the non-crystalline surface being able to identify the adding of lignin in the polymeric matrix and the electrochemical study presented the efficiency of sensor as a conductive material, and its feature of detecting the fungicide (MACIEL et al., 2022).

Colloidal aqueous synthesis and structural characterization of cobalt doped ZnO nanoparticles

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Zinc oxide (ZnO) is an intrinsic binary n-type semiconductor material that belongs to class II-VI, has a direct band gap in the near UV (E_g= 3.37 eV at room temperature), and has an excitonic binding energy of 60 meV. This semiconductor is considered a good host network for doping elements, such as isovalent transition metals (MTs), which have an ionic radius comparable to $Zn^{2+}$and high solubility inside the ZnO matrix [1]. The main strategies established in the literature for obtaining doped ZnO nanoparticles (NPs) are based on co-precipitation or the use of organic solvents. However, colloidal approaches are alternative methodologies to achieve ZnO NPs with lower cost and easy reproducibility [2]. Thus, the present work propose a synthetic method via colloidal chemistry in an aqueous medium to produce cobalt-doped zinc oxide nanoparticles (Co:ZnO NPs). Nitrate salts of zinc and cobalt were used as precursors at a molar fraction according to the expression, $Zn_{1-x}Co_xO$(x= 0.05, 0.075, 0.10), and mercaptosuccinic acid (MSA) as a surface stabilizing agent. The results of X-Ray diffraction (XRD) confirmed the formation of Co:ZnO NPs with an averagediameter variating between 8 - 18 nm. The Transmission electron microscopy (TEM) measurements showed that these Co:ZnO NPs have quasi-spherical morphologies and average sizes of about 9.5 nm. The compositional analyses by energy dispersive X-Ray spectroscopy (EDS) confirmed the doping of ZnO with cobalt. These results suggest thesuccess of the aqueous colloidal route to achieve doped ZnO NPs, which have potentialapplications in several areas of nanotechnology such as sensors, photonics, optoelectronic,photocatalysis, and magnetism.

Acknowledgements: The authors thank the UFPE, CNPq, FACEPE (IBPG-1002-3.03/21), CAPES (88887.832459/2023-00) and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/ 0094/2020).

References:
[1] Singh et al., R.K., Ind. Eng. Chem. Res., 17131 (2019)
[2] van Embden et al., Chem. Rev., 273 (2023)

Construction of a plasmonic biosensor for the detection of Paracoccidioidomycosis

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The localized surface plasmon resonance (LSPR) of gold nanorods (AuNRs) is a label-free biosensing technique known for its sensitivity to changes in the refractive index of the local environment, which is generated by biomolecular interactions around the detection area [1]. LSPR from AuNRs can be used as a sensitive diagnostic tool for important diseases such as PCM. In this study, AuNRs were incubated with different concentrations of the functionalizing agent (2, 4, 6, 8, 10, 12, 14, 16, 18, and 20 mM) and then with different concentrations of the specific protein (2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 25, 27, 30, 50, and 60 μg) to define the optimal conditions for biosensor construction. The UV-Vis spectra of the solutions were obtained for longitudinal band shift analysis (L-LSPR). A significant shift of L-LSPR was observed for all functionalizing agent concentrations tested, with the shift increasing up to the concentration of 8 mM, after which the system reached saturation. Incubating AuNRs functionalized with different protein concentrations did not yield significant differences in L-LSPR shifts up to a concentration of 14 μg. Between 14 μg and30 μg, the shifts were similar, but at concentrations of 50 μg and 60 μg, signs of aggregation were observed. Based on these results, it is recommended to use a concentration of 8 mM of the functionalizing agent and 14 μg of the specific protein for the construction of the proposed biosensor. The biosensor will be tested with fungal samples in the next steps, and sensitivity and specificity analyses will be performed.

Acknowledgements: CDTN/CNEN, SisNano/MCTIC Program, Brazilian Nanocarbon Institute, the Minas Gerais Network of Theranostic Nanomedicine, FAPEMIG, CNPq, CAPES,FINEP,

References:

[1] Cao, J., et al. Gold nanorod-based localized surface plasmon resonance biosensors: A review. Sens. Actuators B Chem, 195, 332-351 (2014).

Contactless conductivity biosensor produced from Printed Circuit Board as durable device for detection of Covid-19 exposure

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The SARS-CoV-2 pandemic revealed the importance of exploring new low-cost materials for point-of-care devices. Given the relevance of the current scenario, the detection and quantification of antibodies can be used to assess the immune response to infection, the disease prevalence, determine a previous infection, as well as help evaluate the immune response to vaccination. Capacitively coupled contactless conductivity detection (C4D) is a promising methodology for these purposes because the reusable electrodes of the C4D sensors are not in contact with the measured fluid; the drawbacks of the polarization effect and electrochemical erosion can be avoided. Moreover, its quick response does not require complex and expensive electronic equipment. In this context, we developed a biosensor for quantifying IgG antibodies to SARS-CoV-2 using the C4D technique and constructed it from a printed circuit board (PCB) as a durable device. The production process used a PCB with a thin 30 μm thick copper layer and a 35 μm PDMS (polydimethylsiloxane) film as an insulating coating, doped with titanium dioxide to explore the variation of the dielectric constant of insulators in the signal gain of a C4D detector. The channels for sample retention were produced using transparent acrylic. Preliminary analyses using sodium chloride (NaCl) demonstrated that the device exhibited good reproducibility (CV ≤ 3.93%, n = 3) and repeatability (CV ≤ 0.40%, n = 10) and a detection limit of 0.34 mmol L-1. An analytical curve was constructed with six antibody concentration levels ranging from 0.05 to 3.30 μg mL-1 (n = 3). The proposed device demonstrated adequate analytical performance, showing a linear relationship between conductivity measurements and antibody concentration.

Copper oxide films as resistive gas sensors

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Copper thin films 20 nm thick were evaporated on mica, and oxidized by three hours at different temperatures (RT, 100, 200 and 300ºC) and room conditions. Raman spectroscopy and UV-Vis spectrometry were performed in all samples. The characteristic Raman shift peaks corresponding to Cu_2O appear clearly in samples oxidized at RT and 100 ºC; slightly,in those oxidized at 200 ºC; and they disappear in films oxidized at 300 ºC. On the otherhand, the peak corresponding to CuO appears in the films oxidized at 300 ºC and 200ºC(slight); and it disappears in samples oxidized at RT and 100 ºC. The UV-Vis spectra show anabsorption peak in 310 nm, for all samples. Alos, the bandgap was determined: values decrease from 1.97 to 1.78 eV as the oxidation temperature increases, similar to others reported for CuO nanostructured samples[1]. The temperature dependence of the resistancewas determined (between 300 and 390 K). The resistance behavior shows a change in theelectrical transport mechanism depending on the oxidation temperature: the temperaturedependence changes from a metallic type in samples oxidized at RT and 100ºC , to a semiconductor type in samples oxidized at 200 and 300ºC. The analysis based on these three characterization techniques show that for oxidation temperatures of 100ºC and RT, the samples are mainly composed by a Cu_2O layer on the Cu film, whereas for 200 and 300ºC, the CuO is the main constituent of the film. Finally, the performance of the samplesas a gas sensor was tested, measuring the electrical resistance of the films in presence ofethanol gas at low pressure (from $10^{-3}$to 10^{3}Pa). The sample oxidized at 300ºC, present a resistance decrease between $10^{-2}$and 10^{0}Pa of up to 40%; while a slight increase appears between $10^{1}$and 10^{3}Pa, the most sensitivity to the presence of the gas.

Acknowledgement

This research was supported by project ANID Fondecyt 1231849

References

[1]M Khan *et al*, Nanomaterials 2020, 10, 1298.

Detection and removal of multiple pesticides fromwater using functionalized composite membrane

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Pesticides are chemicals used to kill larvae, ticks, fungi and weeds, claiming to control the diseases caused by these organisms and regulate the growth of vegetation. The main objective of this project is the development of active membranes based on biopolymer functionalized with xantidrol and ninhydrin to selective detection and removal of the carbaryl and glyphosate pesticides. Characterized by techniques such as BET, FTIR, SEM and TG, Chitosan films were prepared in different percentages of 0.75%, 1% and 1.5% (m/m) in acetic acid. To observe the influence of acid on the physical characteristics of the film, tests were performed altering its concentration of 1% and 2% in the synthesis of the films all contend 0.5% of the glycol polyethylene. For functionalization, 9-xantidrol and ninhydrin of 1%, 2% and 3% (m/m) were used where these compounds were added by direct addition to chitosan gel. To observe the interaction between 9-xantidrol, and ninhydrin and chitosan, UV-vis spectroscopy analysis was performed. Through FTIR and TG, it was observed that the samples of the films functionalized by direct addition there was greater evidence in the spectra obtained and greater resistance to thermal degradation, in which the films of chitosan and xantidrol and ninhydrin, both 2%, are better suited to the proposal. Through UV-vis spectroscopy analysis it was possible to construct calibration curves using composite membrane, showing linear curve with R2=0.996 and R2=0.945 for glyphosate and carbaryl, respectively. The applied methodology proved effective determination of glyphosate and carbaryl and can will be used to verify the removal of the pesticides in water samples.

The authors acknowledge financial support from UFAL, CNPQ, CAPES and FAPEAL

Reference:

[1]LUO, L. et al. Development of an inner filter effect-based fluorescence immunoassay for the detection of acrylamide using 9-xanthydrol derivatization. Sensors and Actuators, Chemical, v. 332. October 2020, p. 129561, 2021.

Detection of “date rape” benzodiazepines in beverages with a silver nanoparticle-enhanced microfluid e- tongue

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The consumption of benzodiazepines (BNZD) has risen significantly during the COVID-19 pandemic, particularly in Brazil, where easy access to these drugs, making them a popular choice for criminal activities such as drug-facilitated sexual assault, also known as date rape. In this study, we present an electronic tongue (e-tongue) device integrated with microfluidics and nanostructured materials to detect three types of benzodiazepines (flunitrazepam, diazepam, and clonazepam) in various beverages, including alcoholic ones. The device is based on the layer-by-layer (LbL) assembly of poly(allylamine hydrochloride) (PAH) and poly(styrene sulfonate) (PSS) and employs silver nanoparticles synthesized through a chemical process to improve the electrical properties of the LbL films. Impedance spectroscopy and Principal Component Analysis (PCA) were used to analyze the results and identify the presence of benzodiazepines in the samples. The e-tongue successfully differentiated between samples with and without the drugs, demonstrating its potential for drug detection. The proposed method could contribute to public health by providing a rapid and reliable tool for monitoring the illegal use of benzodiazepines in beverages.

Detection of ascorbic acid using a 3D printedcolorimetric sensor

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Secondary metabolites are organic compounds produced by plants in response to stress or as a defense mechanism. Although these compounds are not necessary for the plant''s growth or development, they are crucial for its survival [1], as it is the case of ascorbic acid (AA). In this study, we developed a 3D printed colorimetric sensor for detecting AA in liquid samples. The sensor was designed with a hollow microneedle layer and a reservoir layer coated with a composite film of zein and 2,6-Dichloroindondo dye. Upon exposure to AA, the sensor experienced a color change that was quantified using a smartphone to read the RGB patterns of the platform before and after exposure to the analyte. Our preliminary results showed that the platform modified with the 2,6-Dichloroindophenol dye presented a high sensitivity of 47% for AA concentrations ranging from 0.01 to 3 mg mL^{-1}. Furthermore, the platform showed minimal interference with other secondary metabolites, including caffeine, fructose, and H_2O_2, with a maximum sensitivity interference of 12.30%. Our study demonstrates the potential of designing low-cost 3D printed platforms for detecting AA, with applications in various fields, such as in plant and food analysis.

Acknowledgments: This work was supported by FAPESP 2017/20973-4, 2020/14946-7, 2020/22214-6.References:[1] MAHAJAN, M. et al.. Journal of Applied Research on Medicinal and Aromatic Plants, p. 100255, 2020.

Detection of Fe^{3+} Ions with High Sensitivity Using a Photoluminescent Sensor Based on Calixarene Derivative and MOF

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The World Health Organization has established a safe limit of 2.0 mg.$L^{-1}$for iron in drinking water. Atomic Absorption Inductively Coupled Plasma Optical Emission Spectrometry (AAS) is a widely used method for detecting and quantifying metal ions, but it can be expensive and inaccessible. Metal-Organic Frameworks (MOFs) and Calixarenes have both shown potential in detecting metal ions, including iron, in water. A novel photoluminescent sensor has been developed using a post-synthetic strategy to modify the UiO-66-NH_2MOF with calix[4]arene tetracarboxylic acid (CALIX@UiO-66-NH_2). The molecular structure of the calix[4]arene was confirmed using ^1H and ^{13}C Nuclear Magnetic Resonance Spectroscopy (NMR) analysis, and Powder X-ray diffraction (PXRD) was used to identify the crystalline structure of the MOF. The chemical bonding between these two components was confirmed by Fourier-transform infrared micro spectroscopy (μ-FTIR). The CALIX@UiO-66-NH_2presented increased fluorescent emission intensity compared to the MOF and the calix[4]arene and higher sensitivity to Fe^{3+}ions. The detection limit in aqueous media was determined to be as low as 1.6 mg L^{-1}. This study developed a new fluorescent probe, CALIX@UiO-66-NH_2, with enhanced fluorescence emission intensity and greater sensitivity towards Fe^{3+}ions, making it an ideal candidate for sensing applications.

Acknowledgments

This research used facilities of LNLS-CNPEM. The IMBUIA beamline staff is acknowledged for their assistance during the experiments [20221618]. J.D.S.F would like to thank CAPES for a scholarship granted. This work was supported by PRONEX/FACEPE/CNPq (Grant no. APQ-0675-1.06/14).

References

[1] N. Łukasik, E. Wagner-Wysiecka, and A. Małachowska, Analyst, vol. 144, no. 9, pp. 3119-3127, 2019.

[2] Y. Du, X. Li, H. Zheng, X. Lv, and Q. Jia, Anal. Chim. Acta, vol. 1001, pp. 134-142, 2018.

[3] X. Y. Xu and B. Yan, Sensors Actuators, B Chem., vol. 230, pp. 463-469, 2016.

DETERMINATION OF 17α -ETHINYLESTRADIOL IN DISTRIBUTION WATER BY DISPOSABLE ELECTROCHEMICAL SENSOR

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17α-ethinylestradiol is a synthetic hormone, found in female contraceptives, with pharmacological and chemical characteristics similar to estrogens. Among the related problems, the increased possibility of developing vascular diseases stands out, being directly linked to the contamination of effluents, causing problems to the local ecosystem [1]. The development of disposable sensors shows advantages over other systems, showing versatility in combinations, practicality in manufacturing, mass production and relatively low cost, becoming increasingly compatible in analytical applications [2]. Carbon black was chosen because of its conductive character, its nearly spherical particle structure, which aggregate to form a graphite-like structure [3]. The proposed electrochemical sensor was made with conductive ink based on carbon black and glass varnish applied to detect 17α-ethinylestradiol. A compositional study was carried out for a better proportion between the components of the ink, and its electrochemical (cyclic voltammetry) and morphological (scanning electron microscopy - SEM and Fourier transformed infrared spectroscopy - FTIR) behavior. The sensor was subjected to the choice of the best pulse technique (square wave voltammetry), studies of its parameters (amplitude, frequency and step), in addition to studies for choosing the pH. In the construction of the analytical curve, it revealed a linear range from 0.5 umol to 10 umol of 17α-ethinylestradiol and limit of detection (LOD) of 0,2 umol. In this study, the sensor was subjected to water samples from different cities, obtaining recoveries between 85% and 107.6%.

Acknowledgements: CNPq (301796/2022-0), FAPESP (2017/21097-3, 2020/11336-3), and Capes (88887.504861/2020-00)

References:

[1] Torres, N.H; Santos., et al. Chemosphere, Vol. 267, 2021.

[2] Carvalho, J.H.S., et al. Journal of Electroanalytical Chemistry, 2023. 937: p. 117428.

[3] Silva, T.A., et al. Journal of Nanomaterials, 2017.

Determination of catechol with screen-printed paper-based analytical devices made with graphite and glass varnish

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Disposable electrochemical sensors are alternatives in terms of sustainability and low-cost production when compared with some equipment to make quantitative analyses in environmental, medical, and industrially interesting samples. Paper-based analytical devices (PAD) have great advantages and have been considered sustainable for the characteristics of biodegradability [1]. Because of the relatively lower cost, these enable greater accessibility and easier detection of many compounds that require frequent monitoring. An example is catechol (CA) which is on the list of priority pollutants and needs for monitoring in aquatic environments by international organizations [2]. In this regard, a sustainable and low-cost electrochemical sensor was developed, using paper and a conductive ink with graphite powder and glass varnish, as well as its application for the detection of CA. The device was characterized by the techniques of scanning electron microscopy, X-ray diffraction, cyclic voltammetry and electrochemical impedance spectroscopy. Under optimized parameters the proposed sensor was applied for CA detection, which a calibration curve was obtained for the linear range of 1.0 × 10・6 to 1.0 × 10・4 ・mol L・1, in phosphate buffer pH 8.0, with an R2 of 0.99915. The developed sensor is easy to prepare and the results suggest the viability for the detection of CA in real samples.Acknowledgments:Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP), grants# 2019/23342-0, 2017/21097-3, CAPES (001 and CAPES 09/2020 Epidemias 88887.504861/2020-00) , CNPq,(301796/2022-0). References:[1] A.W. Martinez, S.T. Phillips, G.M. Whitesides, E. Carrilho, Diagnostics for the developing world: microfluidic paper-based analytical devices, ACS Publications2010.[2] L.O. Orzari, I.A. de Araujo Andreotti, M.F. Bergamini, L.H.M. Junior, B.C. Janegitz, Disposable electrode obtained by pencil drawing on corrugated fiberboard substrate, Sensors and Actuators B: Chemical, 264(2018) 20-6.

Development of a biosensor for rapid detection of Salmonella in food

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The development of rapid detection techniques for pathogens in foods is still a challenge. Salmonellosis is a foodborne disease and is one of the leading causes of foodborne infections worldwide. In this study, we developed a lateral flow biosensor for rapid detection of Salmonella in food samples [1]. The detection system is based on an immunochromatographic test with gold nanoparticles and antibodies for antigen recognition [2], with results obtained in just 15 minutes after inserting the strip in the sample. The manufactured biosensor has good selectivity and specificity, high sensitivity, with a detection limit of 10^3 CFU/ml, a value below existing commercial tests. The biosensor also showed good repeatability, in addition to being robust, fast and reliable, and could be a favorable alternative for Salmonella screening in foods to ensure food safety.

Acknowledgements:
To Jairo P. Oliveira and my friends at the laboratory. To LANAF, LUCCAR and PPGBioquímica, UFES and FAPES.

References:

[1] Wei, C., Wu, A., Xu, L., Xu, C., Liu, L., Kuang, H., Xu, X. Recent progress on lateral flow immunoassays in foodborne pathogen detection. Journal Pre-proof, Food Bioscience (2023).
[2] Rivas L, de La Escosura-Muniz A, Pons J, Merkoci A. Lateral flow biosensors based on gold nanoparticles. In: In Comprehensive analytical chemistry, 66th edn. Elsevier, Amsterdam, pp 569-605 (2014).

Development of a Lab-made screen-printed systemtreated with CO_2 Laser Pyrolysis for propranolol detection

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Screen printing is a simple method for producing low-cost and feasible electrochemical systems, requiring only an inert substrate, conductive ink, and a screen/stencil with the desired design. The use of CO2 lasers in the fabrication of sensors is an area of research that has generated much excitement due to its potential to change the efficiency of screen-printed systems [1]. In this regard, a new electrochemical device based on conductive ink made of shellac and graphite [2] was developed using acetate sheet as a substrate, which with four electrodes has been carried out, one of which is the reference, the counter and two working electrodes. Morphological and electrochemical characterizations were performed to arrive at optimal parameters, including laser pyrolysis parameters such as laser distance, potential, and burning speed. The optimized system was applied to propranolol, an important drug for cardiac treatments, as well as for the control of hypertension [3]. The results showed that the modified electrodes exhibited increased sensitivity for propranolol, making it a promising tool for the detection of this compound. This lab-made system has the potential to be a cost-effective and efficient method for the detection of propranolol that can be used in various fields, including pharmaceuticals and environmental monitoring. Acknowledgements: CAPES, CNPq (#grant 351261/2022-2) and FAPESP (#grant 2017/21097-3, #grant 2019/23177-0, #grant 2023/00321-3).

[1] ROCHA, Diego P. et al. Chemical Engineering Journal, v. 425, p. 130594, 2021. [2] HENRIQUE, Julia Melo et al. Microchemical Journal, v. 170, p. 106701, 2021. [3] SANTOS, Anderson Marti WONG, Ademar; FATIBELLO-FILHO, Orlando. Journal of Electroanalytical Chemistry, v. 824, p. 1-8, 2018.

Development of an immunosensor based in silica optical fiber to determine ciprofloxacin in wastewater

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Anthropic growth cause the increase of wastewater volume which could impacts environmental matrices such as rivers and lakes [1]. In these wastewaters, so-called pharmaceutical and personal care products are being detected, ranging from hypertensive agents, special care products, stimulants, and antibiotics. Antibiotics, especially ciprofloxacin (CIP), have been widely found in wastewater [2]. In this study, an effective, sensitive, low-cost, and environmentally friendly immunosensor based on optical fibers was developed for CIP analysis in wastewater. A silica fiber of 200 μm diameter, without core, coated with low refractive index polymer was used. A U probe with 30 cm piece was selected and removed 10.00 mm of the coating polymer. The anti-ciprofloxacin antibody was functionalized in the silica fiber and applied in local wastewater samples. The monitored of the optical signal occurred through an evanescent absorbance wave using optical fiber spectrometer. This immunosensor presented a linear range of 0.010 to 1.0×10^4 ng L^{-1}, the detection limit of 3.3×10^{-3} ng L^{-1}, and a quantification limit of 0.010 ng L^{-1} [3]. To validate the developed immunosensor, the data obtained were compared to previous method obtained by Electrochemical Impedance Spectroscopy. At 95 % of confidence through a test-t no significant differences were verified. Both methods presented the same CIP concentration 5.5 ± 0.050 ng L^{-1}.

References

[1] R. Changotra, et al, Journal of Cleaner Production 242, 118544 (2020);[2] I. Michael, et al, Water Research 47, 957-995 (2013); [3] R.S. Lamarca, et al, Analytical Chemistry 92, 14415-14422 (2020).

Acknowledgments

São Paulo Research Foundation (FAPESP grants: #2018/22393-8, #2018/19272-4, #2021/14581-1 and INCT/DATREM 2014-50945-4); CAPES; CNPq; IQ-Unesp-Araraquara and Center for Optics, Photonics and Laser (COPL)-Université Laval.

Development of Screen-Printed Gold Electrodes Modified with Magnetic Nanoflowers for ParacetamolDetection

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Accurate monitoring of drug concentrations, such as paracetamol, is crucial for optimizing clinical outcomes and managing patients'' medication regimens. Electrochemical sensors offer a cost-effective, flexible, disposable, and efficient tool for detecting biomarkers and monitoring drugs [1,2]. However, gold electrodes are relatively expensive, and other alternatives can be chosen for similar purposes. In this study, we describe a method for producing screen-printed gold electrodes on Polyethylene terephthalate (PET) substrates using thermal evaporation. The electrodes were modified with magnetic nanoflowers/poly(3,4-ethylenedioxythiophene) (PEDOT:PSS) for the detection of paracetamol by using electrochemical methods of analysis. The morphology of the nanoflowers was studied using scanning electron microscopy (SEM). The electrochemical sensor performance was evaluated using cyclic voltammetry (CV) and square wave voltammetry (SWV) in the presence of 0.1 mol L-1 phosphate buffer (pH 6.8). The sensor exhibited a linear response for paracetamol concentrations ranging from 2.5 × 10−8 to 5 ×10−2 mol L-1, with a detection limit of 2.2 × 10−7 mol L-1. These results demonstrate thepotential of this simple strategy for developing stable, reproducible, and sensitive electrochemical sensors that could have future applications in virus detection and cancerdiagnosis.

Acknowledgements:
The authors gratefully acknowledge the financial granted by CAPES, CNPq, and FAPESP.

References:
[1] CHUNG, Saeromi et al. Electrochemical carbamazepine aptasensor for therapeutic drug monitoring at the point of care. ACS omega, v. 7, n. 43, p. 39097-39106, 2022.
[2] WANG, Xiaoli et al. A bifunctional electrochemical sensor for simultaneous determination of electroactive and non-electroactive analytes: A universal yet very effective platform serving therapeutic drug monitoring. Biosensors and Bioelectronics, v. 208, p. 114233, 2022.

Difference in fluorescence quenching by GO of anionicand cationic dyes

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A notable property of graphene oxide (GO) is its fluorescence quenching capacity, occurring in two different types: Dynamic (momentary collision interaction) and static (not-fluorescent complex formation). Each of these processes can be described by linear plot using the Stern-Volmer equation. Besides, it is common to observe systems in which both types are present, generating a quadratic Stern-Volmer curve. However, it is required to favor only one type of quenching for some applications, like dynamic quenching for FRET biosensors. For that, to optimize some parameters and characteristics of the medium, fluorophores and quenchers is important to enhance the occurrence of dynamic quenching. In this study, the kinetics of GO fluorescence quenching of four fluorophores with distinct liquid charges are evaluated. For that, the medium parameters like viscosity, temperature and pH have been fixed. Thus, it was observed that process of fluorescence quenching of anionic fluorophores has lower values of association constant (Ka) and positive values for Gibbs free energy (ΔG). Furthermore, in this group the liquid charge is the same of GO in this pH, indicating the repulsive influence of negative charge of both disfavoring the static quenching process. The same conclusion can be taken by observing the relatively high Ka value (2,86) in the interaction of methylene blue (positively charged) and GO. That interaction has a negative ΔG (-2,6 kJ), indicating the favoring of the non-fluorescent complex formation between GO and MB, proving the occurrence of static-quenching process. Therefore, the results show that the distinct electrostatic nature between fluorophore and quencher can be explored to favor one type of quenching and helping to select the optimum dye to construct a FRET biosensor.

Effects of heat treatment on magnetoelastic sensors for application in biosensors

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Magnetoelastic sensors respond to mass variations and, due to the possibility of wireless monitoring, are an excellent alternative for application in biosensors. However, for this to be possible, it is necessary to functionalize the sensor surface to promote the detection of the desired pathogen. However, prior to the functionalization step, it is necessary to cover the sensor surface with layers of chromium (Cr) and gold (Au) to promote corrosion resistance and biocompatibility. Heat treatments (HT) are applied to these sensors to provide better adhesion between the deposited metallic layers and homogenization of the magnetic properties, relieving residual stresses from the alloy manufacturing process and the cutting step to obtain the sensors in the required dimensions. In this context, the objective of this work was to evaluate the effect of HT on magnetoelastic sensors concerning resonance frequency, signal amplitude, and corrosion resistance. For this, sensors produced with Metglas 2826MB alloy were covered with layers of Cr and Au via magnetron sputtering, and four groups of sensors were evaluated: (I) without HT, (II) HT before, (III) HT after, and (IV) HT before and after coating. The HT was carried out at 230°C for 2 hours under a vacuum. The resonance frequency and signal amplitude readings were performed with a network analyzer and an electromagnetic coil system. Corrosion resistance was evaluated by optical microscopy analysis after leaving the sensors in ultrapure water for 48h. It was observed that the heat treatment procedure before and after the deposition of the Cr and Au layers reduced the variance of resonance frequency and signal amplitude. Furthermore, this HT procedure also improved adhesion between the Cr and Au layers with the magnetoelastic substrate, thus exhibiting less corrosion evidence than the other groups.

Electrochemical behavior of dopamine on the surface of electrodes modified with manganese ferrite nanoparticles

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Recently, metal oxide nanoparticles (NPs) have contributed to the development of more sensitive and efficient electrochemical sensors with better selectivity [1]. Electrodes modified with manganese ferrite ($MnFe_2O_4$) will present improved properties promoted by increased electroactive area and high transfer kinetics [2]. In this context, $MnFe_2O_4$NPs were obtained with different properties, and they were applied to prepare carbon paste electrodes (CPE), which were used to dopamine (DA) evaluation. The NPs were obtained by coprecipitation and hydrothermal methods, using $MnSO_4$and $FeCl_3$(1:2) as precursors at 140°C for 24h, NPs were also prepared under similar conditions using β-cyclodextrin as stabilizing agent, this sample was named as CD-$MnFe_2O_4$. The samples showed magnetic behavior, XRD analysis indicated the presence of monophasic manganese ferrite with a crystallite size of 20 nm for $MnFe_2O_4$and 5nm for CD-$MnFe_2O_4$. The NPs possessed high BET area values of 170 $m^2g^{-1}$and 240 $m^2g^{-1}$for $MnFe_2O_4$and CD-$MnFe_2O_4$respectively, the molhability of the NPs surfaces was measured using the contact angle technique, being 110º for $MnFe_2O_4$and 53° for CD-$MnFe_2O_4$characterizing hydrophobic and hydrophilic surfaces respectively. Differential pulse voltammetry measurements of the modified CPE in the presence of DA indicated that the oxidation potential was 0.16V for CPE-$MnFe_2O_4$and was -0.16V for CPE-CD-$MnFe_2O_4$. Analysis of interferents showed that CPE CD-$MnFe_2O_4$is more selective for DA, because no interference from analytes such as ascorbic acid and uric acid, was observed, meanwhile, for the CPE-$MnFe_2O_4$these analytes interfered in the dopamine response. The electrodes showed a good linear range, low detection limit for DA and good sensitivities.

Acknowledgments

The authors thank to CAPES, CNPq, CNANO and LAMAS-UFRGS.

References

[1] S.R. Sabale. Mater. Today. Proc. 33,139-146 (2020).

[2] B.M. Asiabar, M.A. Karimi, et al. Microchem. J., 161, 105745 (2021)

Evaluation of a dynamic method for sensing dissolved oxygen in blood through Fluorescence Lifetime Imaging Microscopy

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The accurate measurement of dissolved oxygen in blood plays a crucial role in monitoring various medical conditions and assessing respiratory function. In this context, a sensor based on coumarin and peptide structures has been explored in this study due to their fluorescent properties and oxygen-responsive capabilities[1]. This sensor operates by monitoring the variation in the photophysical behavior of the sensing system in the presence of dissolved oxygen in blood, using fluorescence lifetimes and their dynamic alterations as parameters, caused by specific interactions with molecular oxygen[2]. The interaction of the analyte with the system causes a change in the dynamic response that is correlated to minimal variations in oxygen concentration, allowing detection of nanomolar levels of O_2in the blood. The response is recorded by a Fluorescence Lifetime Imaging Microscopy (FLIM) microscope, which is available and enables local mapping of the analyte''s concentration variations of interest. This sensor can be applied in areas such as patient monitoring during surgeries, evaluation of respiratory therapy efficacy, and studies of pulmonary physiology. Furthermore, the use of coumarin and peptide structures offers the advantage of being biocompatible, allowing for direct application in the biological environment without adverse effects.

Acknowledgments: CNPq, FAPEG.

References:
[1] Geovany A Souza et al. Adv. Nat. Sci: Nanosci. Nanotechnol. pag 14 (2023).
[2] Sedighi, Mahsa, et al. Journal of Controlled Release. pag 128-145 (2023).

Fabrication and characterization of compact and mesoporous TiO2 thin films for nanobiosensor transducers.

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Glucose sensors are extensively studied and represent a feasible route for introducing innovative sensor designs. This work presents two simple architectures for hybrid organic/inorganic biosensors, with titanium dioxide (TiO2)/indium tin oxide (ITO) thin films as electrodes and glucose oxidase as the catalyser. In the first, compact TiO2 films were fabricated by sputtering and a sol-gel methodology, combined with spin-coating, using titanium ethoxide as the precursor. For the second structure, a self-assembly methodology using pluronic® F-127 as a mesoporous template, together with titanium ethoxide, was used. Self-assembled mesoporous films presented the best cohesion with the substrate/ITO, low microscale crack density as observed by scanning electron microscopy and higher in-plane electrical conductivity. Furthermore, as attested by Raman spectroscopy, the anatase phase has been observed on TiO2 films after synthesis and calcination between 400 and 520°C. Therefore, mesoporous TiO2 thin films present the best biosensor application characteristics due to larger surfaces than compact films. Preliminary results showed, after sensibilization, that all the TiO2 films exhibited an evident change in resistivity in the presence of glucose. Finally, a consistent methodology for fabricating a transducer with a simple and reproducible design is proposed.

Fe_2O_3-based microbial volatile organic compoundssensor

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Microbial volatile organic compounds (MVOCs) are compounds with high vapor pressures and low molecular mass produced by bacteria and fungi [1]. These microorganisms and, consequently, their metabolites can be problematic factors in indoor environments, as they are related to general symptoms of respiratory-tract and eye irritation [2]. Therefore, the development of sensor materials for MVOCs detection is imperative. Sensors based on semiconductor metal oxides (SMOx) are very reported in the literature due to their high performance in detecting gases. Likewise, in the present work, it was produced and characterized the SMOx Fe_2O_3. First, a precursor was synthed, in a Schott bottle, by the mixture of ferric chloride, 1,4-benzenedicarboxylic acid, N,N′-dimethylformamide, and polyvinylpyrrolidone. The mixture was vigorously stirred for 1.5 h at 145 °C. Then, the precipitate was centrifuged and washed with ethanol anhydrous twice [3]. Finally, the precursor was calcined to produce Fe_3O_2. The samples were characterized by X-ray diffraction and field-emission scanning electron microscopy. The sensor test was performed for ethanol, 3-methyl-1-butanol, acetone, 2-butanone, and isovaleric acid at different operating temperatures (200-450 °C). The sensor showed greater selectivity for 3-methyl-1-butanol at 350 °C.

Acknowledgements:

The authors acknowledge São Paulo Research Foundation (FAPESP) (grants 2018/01258-5, 2020/05233-7, 2020/06421-1, 2021/09775-1).

References:

[1] Lemfack, M. C.; Gohlke, B.-O.; Toguem, S. M. T.; Preissner, S.; Piechulla, B.; Preissner, R. Nucleic Acids Res., 46 (D1), D1261-D1265 (2018).

[2] Korpi, A.; Järnberg, J.; Pasanen, A.-L. Crit. Rev. Toxicol., 39 (2), 139-193 (2009).

[3] Cai, X.; Lin, J.; Pang, M. Cryst. Growth Des., 16 (7), 3565-3568 (2016).

Femtosecond laser writing of microstructures indiamond for quantum sensing

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The nitrogen-vacancy (NV) center is a defect in which two adjacent sites in diamond’s tetrahedral lattice of carbon atoms are replaced. One site contains a nitrogen atom instead of carbon while the other is vacant. In its negatively charged state, the NV center gains an extra electron from the lattice, forming a ground state spin system which can be polarized with 532-nm light, even at room temperature. One of the spin states fluoresces much more brightly than the others so that fluorescence can be used for spin-state readout. At the same time, the NV’s electron spin states are sensitive to magnetic and electric fields. These properties make NVs attractive both as a scalable platform for quantum information systems and for high sensitivity electromagnetic field quantum sensors. An integrated optics platform in diamond is essential for both quantum information systems and quantum sensing, where the NV is used as an optically detecting atomic probe. This is because of the ultimate stability and integration provided by monolithic waveguides, in addition to the potential for enhanced optical interaction with NVs. We present results on the femtosecond laser microfabrication of optical waveguides, microfluidics, graphitic pathways and quantum emitters, crucial building blocks for quantum sensing within diamond. We use a microfluidic pump, scanning electron microscopy, confocal fluorescence microscopy, optical waveguide transmission and second order intensity autocorrelation to characterize the performance of these quantum sensing building blocks. In addition, a hybrid fabrication method is introduced whereby ion implantation is used to form NV quantum emitters at nanometric depths at the end facets of laser written optical waveguides. This hybrid fabrication scheme enables development of 2D quantum sensing arrays, facilitating spatially and temporally correlated magnetometry.

Acknowledgment: Research funded by Italian National Project QuantDia (FISR2019_05178)

FLEXIBLE ELECTROCHEMICAL-BIOSENSOR BASED ON POLYPYRROLE TO DETECT THE ONCOGENIC PROFILE OF HPV IN REAL PATIENT SAMPLES

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Cervical cancer is associated with high mortality in women diagnosed with high-risk HPV (human papillomavirus) types. The E6 and E7 genes are used as important oncomarkers. However, current genetic diagnostic methods demonstrate limitations of rapid response with complex protocols. Genosensors are attractive tools that have fast real-time detection [1]. Furthermore, flexible substrates are applied in the structuring of devices, as they provide significant electrochemical and physical properties. In addition, polypyrrole (PPy) and gold nanoparticles (AuNs) provides an increase in the electroanalytical signal [2]. Thus, the objective of this work is the development of a flexible genosensor based on PPy and AuNs for the detection of oncomarkers E6 and E7. For this, a modified flexible ITO was used with the following construction steps: PPy electropolymerization, AuNs electrosynthesis, Cys dripping and primer immobilization. All construction steps and biorecognition tests were characterized by cyclic voltammetry and electrochemical impedance spectroscopy. For biorecognition tests, cervical scraping suspensions were used. Electrochemical changes were observed according to the self-assembly of the genosensor. In the biorecognition tests, there was an increase of ΔI% related to the sample concentration. This indicates the hybridization process. Thus, the developed genosensor can be considered an innovative toolapplied in the oncogenic screening of HPV.

Acknowledgments:
FACEPE, CNPq and CAPES.

References:
[1] Rasouli, E., Shahnavaz, Z., Basirun, W. J., Rezayi, M., Avan, A., Ghayour-Mobarhan, M., ... Johan, M. R. (2018). Advancements in electrochemical DNA sensor for detection of human papilloma virus-A review. Analytical biochemistry, 556, 136-144.
[2] Zare, E. N., Agarwal, T., Zarepour, A., Pinelli, F., Zarrabi, A., Rossi, F., ... Makvandi, P. (2021). Electroconductive multi-functional polypyrrole composites for biomedical applications. Applied Materials Today, 24, 101117.

Gas-Sensing Properties of Zn doped SnO2 Nanoparticles Synthesized via a Nonaqueous Synthesis

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Although semiconducting metal oxide (SMOx) nanoparticles (NPs) have drawn attention as sensing materials, the methodologies available to synthesize them with desirable properties are quite limited and/or often require relatively high energy consumption.[1,2] Thus, we report herein the processing of Zn-doped SnO_2NPs via a microwave-assisted nonaqueous route, at a relatively low temperature (160 °C) and with a short treatment time (20 min). In addition, the effects of adding Zn on the structural, electronic, and gas-sensing properties of SnO_2nanocrystals are investigated. XRD and HRTEM analyses revealed the single-phase of SnO_2, with particle size of 7 nm. XANES measurements revealed the homogenous incorporation of Zn ions into the SnO_2network. From the sensing viewpoint, the results showed that Zn-doped SnO_2NPs were highly sensitive to sub-ppm NO_2levels at 150 °C, presenting good recovery and stability. Also, the Zn-doped SnO_2NPs revealed good stability of their NO_2sensing performance even under ambient moisture. The enhancement in the gas-sensing properties was linked to the Zn ions incorporated that provided more surface oxygen vacancies acting as active sites for the NO_2adsorption on the sensing material.

Acknowledments: We thank the Brazilian financial agencies (FAPESP and CNPq)

References:

[1] M. Niederberger, Acc. Chem. Res., 40, 793-800 (2007).

[2] L. F. da Silva, J.-C. M' Peko, A. C. Catto, et al., Sensors Actuators B Chem., 240, 573-579 (2017).

Gas sensor performance of porous ZnO flowers synthesized by microwave-assisted hydrothermalmethod

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The detection and monitoring of toxic and flammable gases have grown considerably in the last two decades due to increased industrialization. As a result, low-cost, high-sensitivity, and low-energy gas sensors are increasingly in demand. ZnO gas sensors have attracted a lot of attention due to their high chemical stability, non-toxicity, high sensitivity, low cost, and easy fabrication [1,2]. Therefore, the objective of this work is the development of porous ZnO flower sensors for the detection of toxic gases. ZnO was synthesized by microwave-assisted hydrothermal method using zinc nitrate hexahydrate as a precursor. The synthesized powder was thermally treated at 700 °C to create porosity in the material. ByXRD it was identified that the synthesized material is ZnO with a wurtzite structure. SEMimages show that the morphology obtained was porous flowers. Detection measurements forgases demonstrate that the sensor has a high response for $O_3$when compared to $NO_2$and CO, thus indicating that the sensor is more selective for O_3 gas.

Acknowledgments: This research was funded by Sao Paulo Research Foundation's financial support - FAPESP (grants No 2019/22076-5, 2022/03325-7, 2023/00913-8 and 2013/07296-2). The authors are also thankful to the Brazilian National Science Financial Agencies CNPq and Capes.

References:

[1] P. Wang, T. Dong, C. Jia, and P. Yang, "Ultraselective acetone-gas sensor based ZnO flowers functionalized by Au nanoparticle loading on certain facet," Sensors Actuators, B Chem., vol. 288, no. January, pp. 1-11, 2019.

[2] M. Hjiri, F. B. M. S. Aida, and L. E. M. G. Neri, "High Performance CO Gas Sensor Based on ZnO Nanoparticles," J. Inorg. Organomet. Polym. Mater., vol. 30, no. 10, pp. 4063-4071, 2020.

Glyphosate Analysis In Water Samples By Luminescence Spectroscopy Using Lanthanides AsChromophores

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Glyphosate is an active ingredient from herbicides that controls weed and grass development [1]. Its non-selective characteristic harms unwanted plants both in agricultural and nonagricultural zones. When glyphosate is already in the soil, its natural degradation occurs under aerobic and anaerobic conditions by soil microflora [2]. Studies in rodents related to acute exposure to glyphosate and its formulations have shown that the growth was delayed in high-dose females with long-term glyphosate exposure. For high-dose in males, there was a significant increase in the degenerative lens and a liver increase [1]. Consequently, incorrect management that leads to considerable residual spread in the environment, such as in aqueous media, could be dangerous. Based on this, the development of techniques seeking glyphosate analysis by analytical methods has been broadly studied for the past years. However, many methods still require expensive resources, making them impractical. Thus, the present work embraced quantitative glyphosate analysis in water samples by luminescence and UV-Vis absorption spectroscopy, using lanthanides complexes as chromophores, $Eu(DPA)_3$and $Tb(DPA)_3$. With the developed method, glyphosate concentrations could be analyzed based on Brazilian regulations. The developed method had its result compared to a well-established HPLC methodology. Glyphosate complexation has shown photophysical changes after the interaction between glyphosate and metals. One of the main points of this work was the possibility of identifying low concentrations of glyphosate to verify water potability; those concentrations were around 10^{-6}mol. L^{-1}, or 0.5mg. L^{-1}, values recommended by WHO (World Health Organization).

References

[1] S. M. S. Khademi, et al. International Journal of Mass Spectrometry vol. 442, 2019, pp. 29-34.

[2] J. E. Franz, et al. Glyphosate: a unique global herbicide. American Chemical Society Monograph 189. American Chemical Society, Washigton. DC.

graphene oxide quantum dots for fluorescent detectionof Cu^{2+} ions

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Graphene oxide quantum dots (GOQDs) are semiconductor nanocrystals that possess optoelectronic properties which can be controlled by size, molecular structure, and the surrounding physicochemical environment. The fluorescent emission of GOQDs can be quenched upon interaction with metallic ions, making them useful as fluorescent probes in chemical sensors. In this study, we developed an aqueous GOQD suspension for detecting Cu2+ ions. The GOQDs were synthesized by reducing the size of graphene oxide sheets with ammonia and hydrogen peroxide [2], and their structural features were fully characterized using FT-IR and Raman spectroscopy. They absorb at 220 nm and emit at 537 nm, with emission intensity increasing with pH in the range of 4 to 10. At pH 5, the intensity of the fluorescent emission scales linearly (R2 >0.99), but inversely with the concentration of Cu2+ in the range of 1 to 100 micromolar, with a limit of detection of 2.2 micromolar (S/N = 3). It is hypothesized that Cu2+ ions coordinate with carboxylic acid groups in the GOQDs, which are responsible for the fluorescent emission.Acknowledgments: FAP-DF, CAPES, and CNPq.References:[1] P. Zheng and N. Wu, Chem Asian J, vol. 12, no. 18, pp. 2343-2353, 2017.[2] F. Jiang et al., Nanoscale, vol. 5, no. 3, pp. 1137-1142, 2013.[3] E. Dervishi, Z. Ji, H. Htoon, M. Sykora, and S. K. Doorn, Nanoscale, vol. 11, no. 35, pp. 16571-16581, 2019.

Graphen Quantum Dots and Carbon Black nanocomposite for sensitive electrochemical determination of 17-α-ethynilestradiol

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Graphene quantum dots (GQDs) and carbon black (CB) are nanoparticles with electric properties interesting for application in different electrochemical devices, as electrochemical sensors [1]. The present work describe the synthesis and characterization of GQDs nanoparticles and their combination with CB, to modify the glassy carbon electrode and develop a electrochemical sensor (GQDs-CB/GCE) for 17-α-ethynilestradiol (EE2) determination. To synthesis of GQDs, 1,0 g of CA was heated, in vegetal oil bath, at 200ºCfor 30 minutes, then the pyrolysed produt was diluted with 50 mL of a NaOH solution (0,25mol L^{-1}) to obtain a GQDs nanoparticles dispersion. The electrochemical sensor was constructed using a composite of 1,0 mg mL^{-1}of CB, 300 µL mL^{-1}of GQDs dispersion and 1,0mg mL^{-1}of dihexadecylhydrogenphosphate in water. 6,0 µL of the as-prepared composite was dropped on the GCE surface, and dried at 50ºC. Transmission electronic microscopic(TEM) images of the GQDs-CB showed the presence of particulates on two scales, with smaller particles expected to be GQDs and larger particles expected to be CB. UV-vis absorption spectra of the GQDs showed a discreet absorption band with maxima wavelengthat 360 nm, associated with n->π* transitions of residual C=O bonds, and indicates the presence of GQDs in the dispersion. Cyclic voltammograms obtained using GQDs-CB/GCEpresented only a peak current, for EE2, at 0,8 V vs Ag/AgCl (3,0 mol L^{-1}KCl). The currentpeak intensity was 6 times higher than the current response obtained for bare GCE. Theelectroanalytical method was developed using a square wave voltametric technique and were analyzed samples of pharmaceutical formulations and lake water.

Acknowledgements:
To the funding agencies CAPES, UEL Fundação Araucária ((243/2022-PBA) and CNPq ((307505/2021-9).

Reference:
[1] S, Anderson and W, Ademar, Talanta 224, 121804 (2021).

Green electrochemical sensors using CO2 Laser Pyrolysis on dehydrated green leaves substrates forguaiacol detection

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The use of dehydrated green leaves as a substrate for manufacturing electrochemical sensors is an innovative approach that has the potential to provide a sustainable and eco-friendly alternative to traditional sensor substrates. CO2 laser pyrolysis enables the fabrication of precise, complex structures on various substrates, contributing to the development of high-performance, eco- friendly and cost-effective sensors. The green Leaf sensor has been optimized and standardized according to the laser cutter's setting possibilities, such as laser distance, potential and burning speed. In this sense, the leaves used for manufacturing electrochemical sensors were from S. macrophylla and P. Americana, tree species widely and easily found in Brazil [1,2]. The prepared system was used for the detection of guaiacol. Guaiacol It is widely used in medicine due to its potent antimicrobial, anti-inflammatory and analgesic properties. [3]. Indeed, the detection of guaiacol is important in several industries, including pharmaceutical, food, and environmental industries, for example, for quality control of drugs, to indicate the degree of wood smoke flavor in certain foods, or to monitor the level of the compound in air and water, as it can have adverse effects on human health at high concentrations. To this end, the work demonstrates a new system prepared with dehydrated green leaf substrate using CO2 laser pyrolysis for the detection of guaiacol.Acknowledgements: CAPES, CNPq and FAPESP (#grant 2017/21097-3, #grant 2019/23177-0, #grant 2023/00321-3).References:[1] NParks Flora Fauna We [Last updated on 06 April 2023].[2] Persea americana. In: A Guide to Poisonous House and Garden Plants. [s.l.] Teton NewMedia, 2007. p. 209-211.[3] PubChem [Internet]. Bethesda (MD): National Library of Medicine (US), National Center for Biotechnology Informatio 2004-.

Hydrophilic CdTe quantum dots stabilized with cysteamine for sensing of HCO_3^- ion

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Quantum dots (QDs) have aroused interest in the scientific community in recent years due to their distinct optical and electronic properties, being extensively studied for different types of applications. In view of their sensing properties, they have also been employed as sensors for ionic species, especially for heavy metals [1]. Bicarbonate ions (HCO_3^-) play a key role for aquatic life and the distribution of flora and fauna, being an important source of alkalinity and buffering in natural aqueous environments [2]. The detection of anionic species in an aqueous media is a challenge, as the polar nature of water weakens the interactions between sensors andions. Therefore, it is necessary to develop approaches capable of detecting HCO_3^- with high sensitivity. In this work, we used CdTe QDs as a fluorescent sensor for bicarbonate ions. CdTe QDs stabilized by cysteamine (CYA) were prepared in an aqueous media through a one-pot procedure. The CdTe-CYA QDs showed good optical properties and high photostability. The detection of HCO_3^- ions was studied in water, by adding the anions to the QDs suspension. The results showed a gradual enhancement in the QDs' emission intensity under subsequent HCO_3^- additions, demonstrating that CdTe-CYA QDs are sensitive to this anion. Thus, CdTe QDs present the potential to be used as fluorescent sensors to monitor anions in water sources.

Acknowledgements:
The authors thank the UFPE, PGMTR, CNPq, FACEPE (IBPG-1528-3.00/21), INCTAA and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020).

References:
[1] Lesiak et al., Nanomaterials, 2019, 9, 192.
[2] Capella et al., Sensors, 2019, 19, 5528.

impedimetric e-tongue for discrimination and quantification of kombucha-fermented products

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Kombucha is a type of non-alcoholic beverage that is made through the fermentation of sweetened tea using a combination of bacteria and yeast [1]. It is crucial to monitor the fermentation products to ensure that the quality of Kombucha beverages remains consistent. This study demonstrates the use of an impedimetric electronic tongue (IET) to distinguish and quantify kombucha-fermented products such as ethanol, acetic acid, citric acid, and glucose. The IET consists of an array of interdigitated microelectrodes that are coated with layer-by-layer films of different nanomaterials such as gold, cerium oxide nanoparticles, and graphene oxide. By measuring capacitance and resistance values at 1 kHz, the IET can distinguish between kombucha samples of different flavors and brands and monitor the changes in flavor characteristics after opening the sample bottle. Using principal component analysis, the IET was able to quantify ethanol within a linear range of 0.1% to 1% v/v (R2 >0.99). This performance was comparable to the reference method (Anton Paar Alcoholizer 3001) at a 95% confidence level (t-Test). Additionally, the IET was also able to respond linearly to acetic acid, citric acid, and glucose in the range of 2.5-20 g L-1. Overall, the results demonstrate that the IET is a reliable and accurate tool for monitoring the fermentation products of kombucha. Acknowledgments: FAP-DF, CAPES and CNPq. References: [1] N. Barkat et al., Crit Rev Food Sci Nutr 2022, 10.1080/10408398.2022.2069673.

Improved Synthesis and Characterization of Titanium-Modified Silica Nanoparticles using Modified Stöber Method

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Synthesis of silica nanoparticles has been used in a wide variety of areas of knowledge due to the ease and its cost, thus facilitating their potential application in different areas, such as drug delivery systems, electronic devices, biofertilizers, and others. In agriculture, it allows overcoming several obstacles faced in the use of biofertilizers, such as reproducibility, dehydration, stability, andtemperature sensitivity. In this context, the association of nanostructured materials and bacteriathat promote plant growth (BPCVs) can be an efficient tool, increasing the potential of BPCVs as inoculants. The particle can be adjusted through the ratio between the amounts of TEOS and ammonia. However, mesoporous silica nanoparticles are difficult to detect inside plants, so it is necessary to combine them with some other easily detectable chemical element. One method to solve thisproblem was to introduce a detectable chemical element into the nanoparticle synthesis. Inthis work, the synthesis of silica nanoparticles was modified by introducing Ti(IV) isopropoxide (IPT) in the reaction medium. These nanoparticles were prepared with different s by adjusting the molar ratio between TEOS/NH_3 and TEOS+IPT/NH_3 to obtain $SiO_2$and SiO_2@TiO_2 nanoparticles. The aim herein was to evaluate the influence of the TEOSand IPT mixture on the final of the nanoparticles using factorial design techniques for experiments to later monitor the movement of these nanoparticles through the different tissues of the plants. The obtained nanoparticles were submitted to optical, spectroscopic, and structural characterizations and also to incorporation and release tests with dyes.

ACKNOWLEDGEMENTS

This work was partly funded by FAPESP (2013/07296-2) and (2022/10016-0), CNPq (309711/2019-3) and CAPES(88887.641038/2021-00), (88887.643328/2021-00) and (8887.828064/2023-00).

Influence of citrate concentration on AgNPs reductionfor SERS detection of the pesticide 2,4-D

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The environmental pollution concern has been intensified due to consumer demand. New pesticides have been incorporated into daily agriculture to achieve higher production and a shorter time to harvest. In this sense, pesticides can be present as a residual contaminant in our food, soil, and water [1]. Alternatives to detect pesticides in different matrices have been developed and studied, seeking more reproducibility and signal accuracy. Surface- enhanced Raman scattering (SERS) has emerged as an alternative to detect pesticides inlow concentrations exhibiting signal accuracy [2]. However, the detection of certain challenging molecules such as 2,4-Dichlorophenoxyacetic acid (2,4-D) has proven to be difficult due to its highly electronegative nature and carboxylic groups, which makes it less responsive [3]. Herein, the citrate effect on the silver reduction applied in the quantitative analysis of 2,4-D by SERS was evaluated. AgNPs were synthed using citrate as a reduction agent at a concentration of 1% (w/w) ($AgCit_{1.0}$), 0.5% ($AgCit_{0.5}$), and 0.25% ($AgCit_{0.25}$). The decrease of citrate molecules present on the AgNPs surface increases the interaction of the analyte, leading to a limit of detection (LOD) of 1.46 $x10^{-9}$mol/L, while the normal synthesis ($AgCit_{1.0}$) decrease significantly the ability to detect this analyte, leading to a higher limit of detection (1.58 $x10^{-7}$mol/L). In this context, the study of the surface chemistry of plasmonic nanoparticles applied on SERS becomes essential for the efficiency of detection and quantification of the desired analyte.

Acknowledgment

The authors would like to thank the FAPESP [#2018/22214-6, #2020/06577-1 and #2021/13703-6], CNPq, and INCT/INEO for the financial support.

References

[1] Rajput, S. et al. Environ. Dev. Sustain. (2022) 24, 6032-6052.
[2] Rubira, R.J.G. et al. Vib. Spectrosc. (2021) 114,103245.
[3] Kamran, M. et al. J. Mol. Liquids (2019) 273, 536-542.

Influence of Oxygen Concentration in RF-Magnetron Sputtering Deposition Atmosphere on ZnO/CuO Nanocomposite Gas Sensors

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Nanostructured materials based on metal oxide semiconductors (SMOx), such as ZnO and CuO, offer great potential in the development of gas sensors due to their high sensitivity and response and recovery times. However, challenges remain in improving their selectivity and lowering the operating temperatures. The sensing performance of these materials can be tailored by controlling factors such as morphology, particle , defects, and carrier density through the synthesis process[1]. In this study, we explored the effect of varying the deposition atmosphere on the gas sensing properties of ZnO/CuO nanocomposites fabricated using the RF-Magnetron Sputtering technique. The materials were characterized by X-ray diffraction (XRD), UV-Vis spectroscopy, scanning electron microscopy (SEM), Mott-Schottky analysis, and X-ray photoelectron spectroscopy (XPS). Our results demonstrate that the concentration of oxygen (O2) in the deposition atmosphere plays a crucial role in modulating the sensing properties the ZnO/CuO for different gases. This work contributes to the understanding of the relationship between deposition parameters and the sensing performance of ZnO/CuO-based gas sensors, paving the way for the development of more efficient and devices.

Acknowledgements:
This study was funded by the Coordination for the Improvement of Higher Education Personnel - Brazil (CAPES) - Finance Code 001 and FAPESP (Processes 2013/07296-2 and 2019/22076-5) and CNPq (Process 3017231/2021-6).

References:
[1] M. A. Franco, et al, Sensors and Actuators Reports, vol. 4, p. 100100, 2022.

Influence of sample preparation method on Ag colloid aggregation and consequences to carbendazim detection via Surface-Enhanced Raman Scattering (SERS)

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Faced with the impossibility of finding a safe dose for human consumption, the use of the fungicide carbendazim (MBC) was prohibited by ANVISA in 2022. Therefore, the detection and monitoring of MBC is of great importance. Among the existing techniques, SERS has often been used for the detection of pesticides. In this work, it was evaluated the influence of sample preparation on Ag colloid aggregation and, consequently, on MBC detection via SERS. The sample preparation was performed by using standard addition (SAM - successive additions of MBC solution to the same Ag colloid dispersion) and external addition (ESM - a neat Ag colloid dispersion for every aliquot of MBC solution) methods. For SERS measurements, the detection was made at similar concentrations for both methods (1.3x10-6 mol/L). For SAM, the best signal/noise ratio occurs at 4x10-6 mol/L and decreases after this. For ESM, it decreases just after 1.3x10-6 mol/L. In the extinction measurements, for ESM, the appearance of the aggregate band occurs in a concentration close to that by SAM (1.3x10-6 mol/L) with an offset in maximum intensity depending on MBC concentration, suggesting differences in the morphology of the aggregates [1]. In the study of the size distribution by DLS (dynamic light scattering), there is a more pronounced displacement for larger sizes at 1.3x10-6 mol/L for ESM. By SAM, it happens more continuously. For zeta potential measurements of Ag colloid as a function of MBC concentration for both methods, the values become less negative, indicating a probable aggregation of AgNPs, with the chloride ions surrounding the AgNPs being replaced by MBC molecules. In conclusion, ESM and SAM methods showed similar results for SERS in terms of spectra in function of MBC concentration, despite the differences found for DLS and UV-Vis.

Acknowledgements:
FAPESP (2021/02746-6 and 2018/22214-6), CNPq, INCT/INEO and CAPES.

References:
[1] S. Sánchez-Cortés et al. J Colloid Interface, vol. 167, 428-436 (1994)

Influence of the film thickness on the methanedetection of CuO films grown by DC magnetronsputtering

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We present the study of CuO thin films grown using a DC magnetron sputtering. Firstly, Cu thin films were deposited during 1 min onto borosilicate glass substrate using a metallic Cu target. After that, a thermal annealing at 500°C during 2h in air atmosphere were carriedout to obtain the CuO films. Films with different thicknesses were obtained by changing thepower (100, 150, 200, 250 and 350 W) of the power supply. X-ray diffraction (XRD) dataanalysis confirmed the CuO phase formation, without any evidence of extra phases. XRDdata analysis indicate that the crystallite increases from 39 nm to 54 nm as the power source is increased from 100 to 350W, respectively. Furthermore, the residual strain showslarger value for the thinner film, which has been assigned to the structural disorder due tothe strong film-substrate interaction. The unit cell volumes vary in the range 81.36 to 81.24$Å^3$, which is larger than that expected for bulk CuO (81.08 $Å^3$) [1].Cross-section scanning electron microscopy images indicate a growth rate of (3.6±0.2) nm/W and the film thicknessvary from 256 to 1231 nm when the power vary from 100 to 350 W, respectively. Ramanspectroscopy measurements evidenced the presence of only canonical modes associatedwith CuO crystalline phase [2], confirming the results obtained from XRD data analysis. Additionally, UV-Vis spectroscopy measurements revealed a band gap energy (E_g) varyingfrom ~1.74 eV to ~1.41 eV with the power of the power source. That band gap energy reduction trends were assigned to the structural disorder decrease with the film thickness. Tests of gas sensitivity were carried out using methane gas (NH4) at different working temperatures (323K-473 K). Results indicated that the better sensor response was obtainedfor the thinner film. It was associated with the smaller grain for this thinner film in comparison to the others [3]. These results suggest that the CuO thin films are promisingfor potential sensing application.

ISOTHERMAL AMPLIFICATION AND NANOGOLD: GOLD NANORODS AS BIOSENSOR COUPLED TO LAMP TECHNIQUE FOR OPTIMIZATION

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Loop Mediated Isothermal Amplification (LAMP) is a promising genetic material amplification technique that offers advantages such as high specificity, isothermal amplification, reduced reaction time and no need for complex laboratory equipment[1,2]. Gold nanoparticles (AuNPs) are also of interest in the diagnostic innovation scenario due to their excellent performance as biosensors, owing to their unique physicochemical properties[3]. Their versatile surface chemistry allows them to be easiy bioconjugated with a wide range of molecules, making them excellent signal transducers in the construction of biosensors[3]. The objective of this work was to develop a rapid, specific, and sensitive test for the detection of the SARS-CoV-2 virus using biosensors consisting of AuNps conjugated with specific DNA probes that recognize the material amplified by a RT-LAMP reaction and this approach was named iAnG (Isothermal Amplification and Nanogold)[4,5]. RT-LAMP reactions were performed for 15 minutes at 65ºC, using the WarmStart® Colorimetric LAMP 2× Master Mix [NEB #M1804] and N gene as the target. AuNps were synthed as nanorods and conjugated with a probe designed to recognize a specific region of SARS-CoV-2 N gene. The biosensors were titrated with LAMP reaction products and results observed in a UV-Vis Spectroscopy and dynamic light scattering device. UV-Vis analysis showed shifts in the longitudinal plasmon resonance peaks for positive controls, while negative control's sensors had their absorption spectrum disintegrated. In dynamic light scattering, differences in tau parameter was clearly observed between negative and positive samples and graphs showed different patterns for each control. Results showed an efficient proof-of-concept for iAnG test detecting the genetic material of SARS-CoV-2, and it was possible to reduce the RT-LAMP reaction time from 40 to 15 minutes. In conclusion, iAnG technique could be a promising approach for rapid ans sensitive diagnosis.

Lab-made conductive ink for the production of immunosensors for α-synuclein detection in different matrices

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Lab-made conductive inks are interesting alternatives for electrode materials, as they present advantages such as their inherent relative low cost and ease of preparation and use [1]. Among neurological diseases, Parkinson's is one of the most common, which appears with the loss of pigmented dopaminergic neurons in the substantia nigra and accumulation/deposition of α-synuclein in the brain, making its detection of clinical interest for early diagnosis [2]. Here we present the development of a carbon black and poly(vinyl alcohol) glue conductive ink and its application as an electrochemical biosensing of clinical interest analytes. The ink was prepared by mixing the two components in a dual asymmetric centrifuge. After, electrochemical sensors were produced, which were morphologically and electrochemically characterized by techniques such as scanning electron microscopy and cyclic voltammetry. Pd nanoparticles were electrodeposited over the proposed sensor. This procedure was explored and optimized with different experimental designs. The sensors were then further modified with proper and specific antibodies for the analytical detection of α-synuclein, by electrochemical impedance spectroscopy. Two calibration curves were obtained with such material: the first in phosphate buffer, where the linear behavior was acquired between the concentration of 1.5 and 15 μg mL^{-1}, with a calculated LOD of 0.13 μg mL^{-1}; and the second one, where the linear range was observed from 6.0 to 100.5 μg $mL^{-1}$of a-synuclein, with a LOD = 1.31 μg mL^{-1}. The ink is easy to prepare and use, and the produced devices demonstrated attractive results for the clinical diagnosis of neurological diseases related to α-synuclein, both in simple and complex sample matrices.

References:
[1] I. A. A. Andreotti, et al., Journal of Electroanalytical Chemistry, 840(2019).
[2] W. Poewe, et al., Nature reviews Disease primers, 3(2017).

Layer-by-layer film containing $CoFe_2O_4$ nanocrystals as sensing units in electrolyte-insulator-semiconductor devices for H_2O_2 detection

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Nanostructured materials have demonstrated significant potential for applications in the field of (bio)sensing. Among these, the capacitive electrolyte-insulator-semiconductor (EIS) sensor is a suitable field-effect device for nanostructured films integration as sensing units [1,2]. In this work, we investigate the fabrication of a hybrid nanostructured film using the layer-by-layer (LbL) technique combining cobalt ferrite ($CoFe_2O_4$) nanocrystals dispersed in poly(vinylpyrrolidone) (PVP) and poly(amidoamine) (PAMAM) dendrimer. LbL films containing a PAMAM/PVP-$CoFe_2O_4$ architecture with different bilayers were fabricated onto EIS chips of Al/p-Si/SiO_2. The morphology of the films was characterized by atomic force microscopy (AFM), and the sensing performance toward H_2O_2 detection evaluated by capacitance-voltage (C/V) and constant capacitance (ConCap) measurements. The surface of a 3-bilayer film observed in the AFM images exhibited a homogeneous distribution with random distribution of spherical-shaped agglomerates identified as Co_2FeO_4 nanocrystals with diameters ranging from 20 to 40 nm. Films with different bilayers were also analyzed by AFM microscopy, showing that for higher bilayers a homogeneous pattern of nanocrystal distribution was not observed. Analyzing the electrochemical and morphological characteristics of the films, we found an optimized system. The results indicated the best performance toward H_2O_2 exhibited by 3-bilayer EIS-(PAMAM/PVP-$CoFe_2O_4$) sensor, which presented a sensitivity of 26.5 mV decade^{-1} and a low limit of detection of 157 µM. The possible overall mechanism that may explain the operating principle of the EIS-$CoFe_2O_4$ sensor is assigned to the catalytic activity of $CoFe_2O_4$ nanocrystals toward H_2O_2 [3]. The set-up achieved in this work, a field-effect sensor for H_2O_2 detection, appears as an alternative to conventional amperometric H_2O_2 sensors.

Low-cost and portable colorimetric biosensor is produced for the diagnosis of viral diseases as a point-of-care tool

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The frequent outbreaks of infectious diseases around the world have presented significant challenges to health systems, requiring the development of rapid and accurate diagnosis devices for effective monitoring and control in remote areas [1]. In this study, a low-cost and portable biosensor based on cellulose paper with specific antigen (IgG) conjugated to gold nanoparticles (AuNPs) is produced for the diagnosis of viral infections, using a smartphone as a detector. AuNPs were synthed and characterized using specific techniques (UV-Vis, FTIR, TGA, and MET). A 23 factorial design was then employed to optimize the conditions for conjugating AuNPs with the IgG antibody (AuNP@IgG). Three factors were evaluated: (1) concentration (50-320µg.mL-1), temperature (4-37°C), and pH (5-9). As results, the AuNPs were successfully obtained with particle of ~90 nm, and spherical morphology, as indicated by MET imaging. The response surface analysis indicated the optimal conditions for the IgG conjugation to AuNPs, with a concentration (360 µg.mL-1), temperature (25ºC),and pH (7.4). Currently, the impregnation studies of AuNP@IgG onto cellulose paper andthe optimization detection system using smartphone (PhotoMetrix App) are being investigated, and the findings will be presented at the event.

Luminescent gold nanoclusters obtained by pulsedlaser ablation in water: modulation of the photoluminescent response by the interaction withheavy metal ions

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Sensitive sensors for the detection of heavy metal ions (HMI) in water are in high demand, due to the danger they represent for both environment and human health. We report different simple, green and cheap experimental methods for the separation of luminescent gold nano-clusters (AuNCs) from a pristine colloidal dispersion of gold nanoparticles (AuNPs) obtained by pulsed laser ablation (PLA) in water. Independently on the separation technique, the bleached material present both UV and blue emission, a concentration less than 1 ppm, a negative charge, and dimensions ranging from 0.5 to 2.5 nm, as evidenced by high resolution electron microscopy. The bleached AuNCs colloids were used as luminescent chemosensors for the detection of Cd^{2+}, Pb^{2+}, $Hg^{2+}$and methyl mercury $CH3Hg^{+}$ions in water. The intensity of blue emission decreased (no more than 10%) and saturated at concentrations higher than 20 ppb for all the heavy metal ions tested. In contrast, the UV band emission was remarkably affected in the presence of Hg^{2+}ions, thus leading to signal variations for concentrations well beyond 20 ppb (the concentration at which saturation occurs for other ions). The limit of detection for $Hg^{2+}$is about 3 ppb (15 nmol/L), and the photoluminescence intensity diminishes linearly by about 75% up to 600 ppb. The results are interpreted based on the ligand-free interaction, i.e., the metallophilic bonding formation of $Hg^{2+}$and Au^{+}oxide present on the surface of the UV-emitting nanoclusters [1].

References

1) Tahir et Al., "Quenching of the Photoluminescence of Gold Nanoclusters Synthed by Pulsed Laser Ablation in Water upon Interaction with Toxic Metal Species in Aqueous Solution", Chemosensors 2023, 11(2), 118; https://doi.org/10.3390/chemosensors11020118

MIL-88b-NH_2 and MIL-82 as a candidates for modify carbon paste electrode for quantify Cu^{2+} in ethanolic media

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Metal-organic frameworks (MOFs) are porous coordination polymers designed based on the variety of the metal coordination environment and the nature of the organic ligand to form a network [1]. These materials have attracted research interest due to their promising applications. However, their use as electrochemical sensors is rare due to their poor electrical conductivity. In this sense, MOFs have been tested as carbon paste electrode (CPE) modifiers. The sp^2 carbons and the presence of six-membered aromatic rings in the graphite structure allow electrical conductivity when forming composite. This is provided by structural changes in the surface area of MOF. The sensing capability of a Fe^{III}OFs was used for detecting of Cu^{2+} in ethanol (EtOH). Cu^{2+} can be incorporated into EtOH due to the intensive use of pesticides in sugarcane plantations. PXRD and FTIR were employed to characterize the synthesized MIL-88b-NH_2 and MIL-82 structural features. According to the results, both MOFs were successfully obtained. MIL-88b-NH_2/CPE was chosen to be tested as a Cu^{2+} sensor in EtOH based on CV result. Thereafter, SWASV was used to quantify the Cu^{2+}. The analytical signal of the electrode showed good stability (RSD = 5.2%). The peak current of MIL-88b-NH_2/CPE gradually increased with the addition of Cu^{2+}. Furthermore, r and R^2 were equal to 0.9993 and 99.85%, respectively, with a LOD = 0.020 μmol L^{-1}. Our findings indicate MIL-88b-NH_2 modified CPE in an appropriate sensor for evaluating EtOH by Cu^{2+}.

Acknowledgements:

The authors are grateful to CAPES, LPDE, and LabPetro for financial and technical support.

References:

[1] Hanikel N, Prévot, MS and Yaghi OM, Nat. Nanotechnol. 15, 348-355 (2020)

MIP-based electrochemical sensor for COVID-19 diagnosis in saliva

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This work developed a sensor for diagnosis of COVID-19 using screen-printed electrode (SPE) electrochemically modified by MIP using pyrrole (Py) as monomer and the spike protein (S-protein) as template, named MIP-PPy. The number of cycles used in the electropolymerization and the monomer and template concentrations were optimized.[1] Template removal was performed using a 10% (w/v) HAc solution and 0.2% (w/v) sodium dodecyl sulphate for 2 h. The thickness of the optimized sensors was estimated by SEM analysis. A pre-concentration step applying +0.6V potential for 1 min was performed to achieve detectability on a fentrogram scale. The sensor was also subjected to tests of specificity, stability over 21 days, and analysis of clinical samples. The sensor response for S-protein was done by indirect determination using probe $[Fe(CN)_6]^{3-/4-}$ by differential pulse voltammetry technique. The best conditions were defined as pyrrole at 0.3 mol/L, S-protein at 40 μg/mL, and 4 cycles. SEM images confirmed the formation of the polymeric film since MIP-PPy showed a greater thickness compared to the NIP-PPy (non-printed sensor) and the bare SPE. Under optimized conditions, rebinding assays after the pre-concentration step using 100 fg/mL of S-protein in PBS (pH 7.4), showed a decrease in probe current when using MIP-PPy regarding the NIP-PPy ones. Such behavior reveals the selective interaction of the S-protein with the imprinted cavities. The sensor showed good specificity. The electrochemical response of MIP-PPy reduced 15% of its initial value after 14 days, showing intermediate stability. Sensor validation with saliva samples showed agreement with RT-PCR results. Therefore, the developed sensor proves to be a great alternative method to RT-PCR, featuring good detectability, fast analysis time, specificity, portability, and simplicity of synthesis.

Acknowledgments: CAPES, CNPq, UEL, IFPR.

References:

[1] Q.Zeng, X.Huang, M.Ma, Int. J. Electrochem. Sci., 12, 3965 (2017).

MOF-derived Co_3O_4 produced by a microwave-assisted hydrothermal method for 2-Butanone sensor

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2-Butanone is one of the most essential ketones in the industry due to its use as a solvent in paints, pharmaceuticals, adhesives, cosmetics, etc. [1]. Besides, patients with lung cancer exhale 2-butanone in their breathing [2]. Therefore, a 2-butanone sensor could be applied in indoor air quality monitoring and non-invasive diagnosis of diseases. In this work, we developed a synthesis of Co_3O_4 using the ZIF-67 precursor with a typical method [3] and applied heat with a microwave (2,5 GHz, 800 W) to speed up the synthesis process. Firstly, a mixture was made using an aqueous solution of 0,45 g of $Co(NO_3)_2.6H_2O$ and 5,54 g of 2-methylimidazole. The resulting solution was stirred, placed in a polytetrafluoroethylene autoclave, and heated at 100 °C for 30 min in the microwave. Then, the purple precipitate of ZIF-67 was centrifuged, washed with ethanol, and dried at 80 °C overnight. ZIF-67 was calcinated at 300 °C for 1 h at a heating rate of 5 °C min^{-1} to produce the Co_3O_4. The sampleswere characterized by XRD, TGA and FESEM. The sensor measurements were carried out atan operating temperature range of 250-450 °C with 100 ppm of 2-butanone, methanol, 2-nonanone, benzene, toluene, and m-xylene. The Co_3O_4 sensor showed a higher response for 2-butanone.

Acknowledgements:
LSQA - Unesp, Embrapa, CAPES (88887.694732/2022-00), CNPq (311453/2021-0) and FAPESP (2020/05233-7 and 2020/06421-1).

References:
[1] Hoell, D.; Mensing, T.; Roggenbuck, R.; Sakuth, M.; Sperlich, E.; Urban, T.; Neier, W.; Strehlke, G. Wiley-VCH Verlag GmbH Co, 209-212 (2009).
[2] Vioto, G. C. N.; Perfecto, T. M.; Zito, C. A.; Volanti, D. P, Ceram. Int.vol. 46(14), 22692-22698 (2020).
[3] Lü, Y., et al. ACS Applied Materials Interfaces, 6(6), (2014).

MOF-derived ZnO/Co_3O_4 n-p heterojunction using an ultrasonic spray method for 3-methyl-1-butanol sensor

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Microbial Volatile Organic Compounds (MVOCs) are produced during the metabolism of fungi and bacteria, which causes several health problems in humans [1]. 3-methyl-1-butanol (3M1B) is an MVOC produced by Pseudomonas spp. and a spoilage indicator of seafood [2]. Detecting the 3M1B is extremely important to bring a better quality of our food. The heterojunction n-p of ZnO and Co_3O_4 was produced from Metal-Organic Framework (MOF). In this work, we developed a synthesis of ZnO/Co_3O_4 using ZIF-8/ZIF-67, adapting a typical method [3]. Firstly, 151 mg of ZIF-8 was dissolved in deionized water, stirring for 30 min, and 62,4 mg of 2-methylimidazole was added. After that, a mixture was made using 5,2 mg of $Co(NO_3)_2.6H_2O$ in an ultrasonic spray. The resulting solution was stirred for 6 h, centrifuged, washed with ethanol, and dried at 80 °C for 20 h. Then, the ZnO/Co_3O_4 was produced by calcinating the ZIF-8/ZIF-67 at 450 °C for 2 h at a heating rate of 2 °C min^{-1}. The samples were characterized by FESEM, XRD, and TGA. Sensor measurements were conducted at different operating temperatures with 100 ppm of 3M1B, 2-butanone, 2-nonanone, m-xylene, toluene, methanol, and benzene. Finally, the sensor showed a higher response for 3M1B at 350 °C.

Acknowledgements:
LSQA - Unesp, Embrapa, CAPES (88887.694732/2022-00), CNPq (311453/2021-0) and FAPESP (2020/05233-7 and 2020/06421-1).

References:

[1] Korpi, A.; Järnberg, J.; Pasanen, A.-L. Crit. Rev. Toxicol., 39(2), 139-193 (2009).
[2] Fan, Y.; Schneider, K. R.; Sarnoski, P. J. Food Chemistry: X, v. 14, (2022).
[3] DOAN, T. L. H. et al. Sensors and Actuators B: Chemical, v. 348, (2021).

Multivariate optimization of electrochemically reduced graphene oxide films synthesized from electronic waste for the voltammetric determination of β-estradiol

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The use of reduced graphene oxide as an electrode modifier material is increasing due to its high surface area and electrocatalytic activity [1]. Conditions of electrochemical reduction are crucial to control the extent of the process. Total reduction can be unwanted because itcan increase the background current and decrease the sensitivity due to the reduced number of oxygenated groups [2]. In this study, factorial design was applied to promote the multivariate optimization of the electrochemical reduction of GO films to enhance the voltammetric response of β-estradiol hormone. GO was synthed by the modified Hammers method using graphite recovered from discharged batteries. Cyclic voltammetry was used to carry out the electrochemical reduction of GO films, which were prepared by the drop casting method using a glassy carbon electrode (GCE). The factorial design used was quadratic with three variables at three levels, as follows: scan rate (ν = 10, 50 and 90 mV s^{-1}), number of cycles (5, 10 and 15) and GO concentration at the aqueous dispersion (0.5, 1.25 and 2.0 mg mL^{-1}). Data analysis was performed in Statistica® software. The matrix with parameters combinations generated 11 assays. The voltammetric profile of the $Fe(CN)6^{3-/4-}$redox probe and the anodic peak current of β-estradiol were recorded and usedas the factorial design response. Thus, it was possible to relate the rate of film reduction with the voltammetric response of β-estradiol. Scan rate was the parameter that influenced the most, as well as its interaction with GO concentration. The optimal conditions were ν = 90 mV$s^{-1}$and c(GO) = 0.5 mg mL^{-1}. On the contrary, the number of cycles did not influence significantly, which allowed the choice of the lowest value (5) for subsequent studies.

Acknowledgements

FAPEMIG (APQ-00083-21; RED-00144-22), CNPq (405828/2022-5) and CAPES.

References

[1] Z. Zhang, et al., Electrochim. Acta, 139, 232-237, 2014.

[2] D. Karačić, et al. Electrochim. Acta, 410, 140046, 2022.

Nanobioelectrode based on polypyrrole film and gold nanoparticles applicable to the diagnosis of acute promyelocytic leukemia

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Hematological neoplasms, commonly called liquid tumors, constitute a heterogeneous group of pathologies. In acute promyelocytic leukemia, the PML/RARα chimeric oncogene is a striking feature, resulting from balanced reciprocal translocation involving the PML gene and the retinoic acid alpha receptor. Although the techniques commonly required in clinical practice prove to be effective for the diagnosis of the PML/RARα fusion gene, they have important limitations. Among these factors, the extensive analysis time and high costs can be highlighted [1]. The aim of this study is to develop a nanobioelectrode based on conductive polymer and gold nanoparticles applicable to the diagnosis of the PML/RARα chimeric oncogene. To characterize the construction stages of the sensing platform, the techniques of cyclic voltammetry and electrochemical impedance spectroscopy were used. In the last stage of platform assembly, through the immobilization of DNA sequences on the surface, it was possible to obtain a modified electrochemically active area for the biorecognition assays [2]. Upon exposure of the genosensor to positive plasmid samples, a decrease in the anodic and cathodic peak currents was observed, as well as an increase in resistance to charge transfer. Thus, the developed genosensor proved to be an efficient tool for identifying the PML/RARα oncogene and monitoring patients during treatment.

Acknowledgements:

CNPq, FACEPE, CAPES and PROPESQI/UFPE.

References:

[1] Chupradit, S., Pattanakul, P., Panya, A., Chailapakul, O., Rodthongkum, N. (2022). Various types of electrochemical biosensors for leukemia detection and therapeutic approaches. Analytical Biochemistry, 114736.

[2] Zhang, Z. Y., Li, W., Xie, Y. Q., Liu, Z. Y., Zhang, X. X., Li, Q. Y., Zhao, J. Y., Li, C. Y., Li, L. (2021). Efficient determination of PML/RARα fusion gene by the electrochemical DNA biosensor based on carbon dots/graphene oxide nanocomposites. International Journal of Nanomedicine, 16, 3497.

Palladium deposition on conductive ink of carbon nanotubes for the construction of an electrochemical immunosensor

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Among the new technologies capable of assisting in the determination of biomarkers of various diseases, electrochemical immunosensors stand out. Such devices gain notoriety in the field of medicine due to the possibility of responses in a shorter time, high sensitivity and aid in diagnosis in the early stages[1]. An alternative for the manufacture of these devices are conductive inks, contributing to a system with different architectures, relatively low cost and possible to be manufactured in the laboratory[2]. Thus, in this study we present the first results obtained for an electrochemical sensor of conductive carbon nanotube ink, deposited on a paper substrate. A study was carried out to waterproof the paper, modifying it with beeswax. Palladium deposition after making the sensor is a study that precedes immunosensor immobilization stages for biomarkers of neurodegenerative diseases. Electrochemical (cyclic voltammetry and electrochemical impedance spectroscopy) and morphological (scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR)) characterizations were used. The concentration of the beeswax/hexane solution for waterproofing the paper was 1g/100 mL. In the SEM images, it was possible to observe a layer capable of covering the pores of the paper is observed, guaranteeing the waterproofing of the substrate. Also, the proposed sensor was modified with Pd particles, which occurred after electrodeposition of the metal by chronoamperometry. The time studied for electrodeposition ranged from 1 to 5 minutes, with 5 minutes being chosen as ideal. To stabilize the particles on the surface, cyclic voltammetry at 50 mV $s^{-1}$was applied. This paper analytical device is an alternative for the development of electrochemical sensors and biosensors.

[1] Oliveira, G.C.M.d., et al. Biosensors and Bioelectronics, 2020. 152: p. 112016.
[2] Carvalho, J.H.S., et al. Journal of Electroanalytical Chemistry, 2023. 937: p. 117428.

Physical-chemical characterization of biochar from cupuassu bark (Theobroma grandiflorum) for application as a soil conditioner

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Abstract: Biochar is the product of pyrolysis of renewable biomass, with low oxygen availability. This material is very porous, rich in carbon and contains a large amount of organic structures and inorganic elements. The chemical composition of the ash fraction consists of macro and micronutrients for plants and improves soil fertility conditions. The objective of this research was to carry out the physical-chemical characterization of the cupuaçu bark biochar, identifying its physical properties favorable to its use as a soil conditioner. The *Theobroma grandiflorum* peels used as raw material was purchased from farmers and fairgrounds in the municipality of Parintins. The synthesis of biochar for physicalchemical and morphological characterization was carried out using the activating agent phosphoric acid (H_3PO_4) in a ratio of 1:1 by mass. The physical and chemical characterization by X-Ray Diffraction Technique (DRX) was performed at Federal Institute of Amazonas, Campus Manaus Centro and the morphological one by Scanning Electron Microscopy (SEM) at INPA's Optical Microscopy Laboratory. It can be concluded that the lower temperature in the fast pyrolysis influences the formation of larger pores. This may indicate that the raw material of the samples was thermally decomposed during the pyrolysis process, as expected, and that the obtained biochar has a high capacity for nutrient and water retention.
Keywords: biochar, nutrient, retention, soil conditioner, water.

Introduction

Biochar is a product of the thermal decomposition of organic materials under low oxygen availability. The chemical composition of the ash fraction consists of macro and micronutrients for plants and improves soil fertility conditions. Biochar can be obtained from different plant species and its application as a soil conditioner has caused an increase in crop productivity in degraded soils (LEHMANN; JOSEPH, 2009 apud RIBEIRO, 2016). The objective of this work is to characterize physically and mologically the biochar from *Theobroma grandiflorum* peels evaluating its application as a soil conditioner in the development of lettuce seedlings

Experimental Procedure

The *Theobroma grandiflorum* used as raw material was purchased from farmers and fairgrounds in the municipality of Parintins. The synthesis of biochar for physical-chemical and morphological characterization was carried out using the activating agent phosphoric acid (H_3PO_4) in a ratio of 1:1 by mass. The procedure consisted of impregnating the cupuaçu peels with activanting agent under constant agitation for 5 h and then, dried in an oven at 105 °C for 12 h. The resulting material was carbonized and activated in porcelain crucibles. The procedure was carried out in a muffle furnace with the following heating steps: 115 °C for 30 min, 360 °Cfor 30 min and 500 °C for 1 h. Biochar samples were synthesized by changing the temperatureof the last heating step to 400 °C. The obtained biochars were washed with distilled water at 80 °C. After washing, all samples were dried at 105 °C for 12 h. All samples were obtained in triplicate. The physical and chemical characterization by X-Ray Diffraction Technique (DRX) was performed at Federal Institute of Amazonas, Campus Manaus Centro and the morphological one by Scanning Electron Microscopy (SEM) at INPA's Optical Microscopy Laboratory.

Results and Discussion

Fig. 1 presents the X-ray diffractograms obtained for the biochar samples synthesized at 400°C and 500°C. It is possible to observe that the samples have an amorphous structure, characterized by the wide peak in the 2θ position from 10° to 30° C. In the case of amorphous materials, the diffraction patterns do not produce narrow peaks as in crystalline materials, but wider peaks, because the arrangements of atoms are irregular (Kahn, 2007). The amorphous peak is attributed to the presence of carbon.

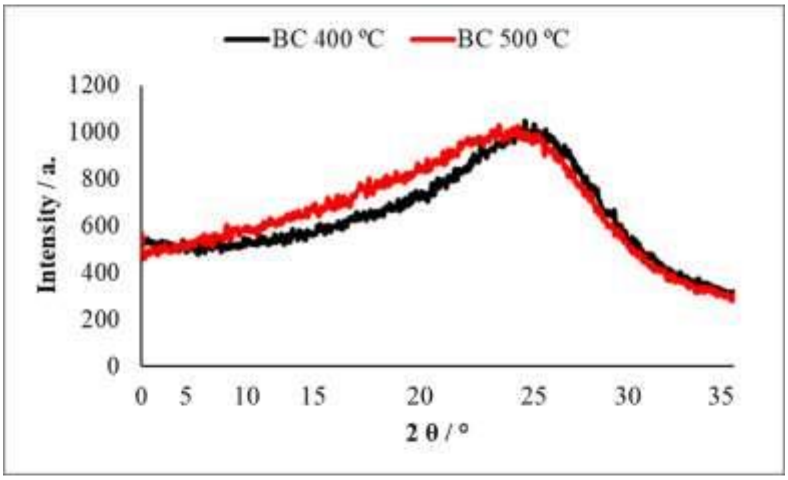

Figure 1: Biochar diffractogram synthesized at 400°C (black curve) and 500°C (red curve).

The micrographs of biochar synthesized at 400°C and 500°C, Fig.2 (A, B, C and D), show the morphology of the material. It is possible to identify that the surfaces are porous. Figures 2 A and C corresponding to the biochar synthesized at 400°C show larger pores than the biochar synthesized at 500°C. It can be concluded that the lower temperature in the fast pyrolysis influences the formation of larger pores. This may indicate that the raw material of the samples was thermally decomposed during the pyrolysis process, as expected, and that the obtained biochar has a high capacity for nutrient and water retention.

At 45 DAS it was verified that the height of the lettuces presented very close values and the widths had significant differences. The lettuce leaves treated with biochar had greater widths than the control and among the proportions used, the dose of 30% provided wider leaves. This can be attributed to the ability of biochar to help retain nutrients. Júnior et al. (2012) verified that biochar, produced in a masonry oven, at a dosage of 15% to the substrate, was a good alternative as a conditioner in the production of lettuce seedlings.

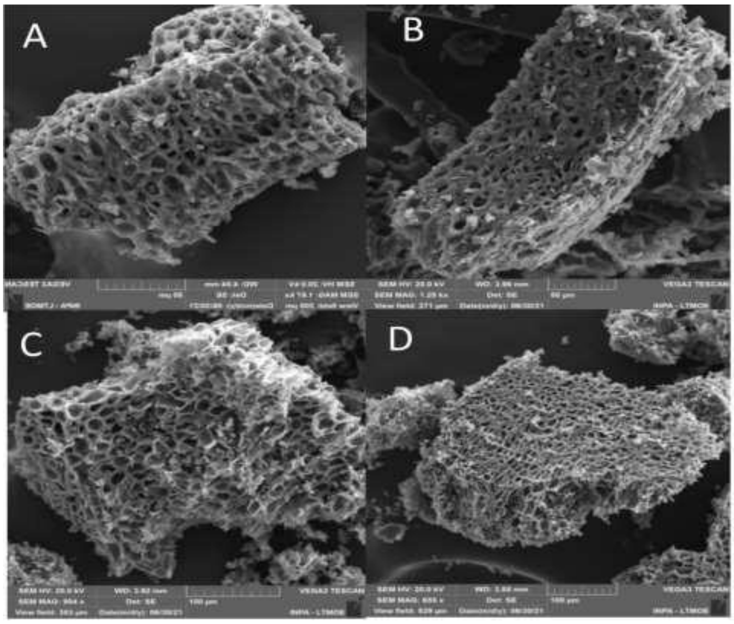

Figure 2: Micrographs of biochar synthesized at a temperature of 400°C (A and C) and 500°C (B and D).

The performance of lettuces in relation to the number of leaves as a function of the BC showed a difference only in the proportion of 30% of biochar, the other proportions had the same number of leaves as the control, as shown in Tab. 1. Regarding the larger leaf, it was also detected that the soil with 30% of biochar showed better growth whose look reached a length of 15.5 cm, showing a superiority of 2 cm over the control. Cavalcante et al. (2012) detected the positive influence of biochar on the formation of yellow passion fruit seedlings as the increase in the proportion of biochar in the substrate up to a concentration of 50%.

Conclusions

The biochar produced by hand with *Theobroma grandiflorum* bark in a proportion of 30% showed better results than the other proportions in relation to height and number of leaves. The other proportions had equal and lower results than the control. Thus, biochar from *Theobroma grandiflorum* bark can be a viable alternative as a material for improving the substrate in the production of vegetables. To confirm the results, further studies are needed.

Acknowledgments

Federal Institute of Amazonas (IFAM) for financial support, to the INPA Electronic Microscopy Laboratory and to the Analytical Center of IFAM Campus Manaus Centro for technical support and to IFAM Campus Parintins.

References

[1] CAVALCANTE, L. et al. Biochar no substrato para produção de mudas de maracujazeiro Amarelo. Revista de la Facultad de Agronomía, 111: 41-47, 2012.
[2] JÚNIOR, B. H. M. et al. Produção de mudas de jiló em substrato condicionado com Biochar. Comunicata Scientiae 3(2): 108-114, 2012.
[3] PETTER, A.F et al. Biochar como condicionador de substrato para produção de mudas de alface. Revista Agrarian, 5:243-250, 2012.

Preparation of mango (*Mangifera indica* L.) waste flour for the application of Cu^{2+} removal in an aqueous medium by adsorption process

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Contamination of water by Cu2+ harms the environment and human health.[1] Adsorption reduces the concentration of these ions.[1,2,3] The material most commonly used in adsorption is activated carbon, but this can be replaced by biosorbents, materials derived from agroindustrial waste biomass that are available, accessible, renewable, and recyclable.[1] The flour from the residue of mango (Mangifera indica L.) is a biosorbent capable of adsorbing Cu2+.[1] This study aimed to verify the efficiency of Cu2+ adsorption using mango waste flour. The preparation of MWF consisted of drying the cores and seeds of Tommy mangoes (80°C, 48 hours), crushing, sifting, and treatment with HCl (0.05mol.L-1, 10minutes);[1,3] MWF was characterized with FTIR and point of zero charge;[1,3] The adsorption studies were conducted at room temperature, pH 4 and agitatio [3] The parameters to determine the optimal adsorption conditions were adsorption time and MWF ma [3] The final concentration of Cu2+ was determined by complexometric titration. FTIR identified organic groups that interact with Cu2+;[3] The point of zero charge (PZC) determined that at a pH 4.3, the charge on the MWF surface is neutral; thus, at a pH lower than 4.3, the charge is positive and interacts with anions, and at a pH greater than 4.3, the charge is negative and interacts with catio [1] The optimal adsorption conditions were 30minutes and 0.1g of MWF; The linearized Freundlich and the non-linearized Redlich-Peterson adsorption isotherm models were found to be more suitable for this adsorption, suggesting a multilayer adsorption mechanism of MWF;[3] The maximum percentage of Cu2+ removal was 77.48%. Thus, this adsorption process using MWF has the potential for removing Cu2+.

Acknowledgements:
UNESP

References:

[1] R. NADEEM et al., Saudi Chemical Society v. 19, p. 23 (2015)
[2] C. SAMPAIO et al., Environmental Science and Pollution Research v. 26, p. 5588 (2019)
[3] E. LEANDRO-SILVA et al., Matéria v. 25 (2020)

Production and Characterization of conductive ink based on commercial glue and graphite powder for the manufacture of screen-printed electrochemical sensors.

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The production of disposable screen printed electrochemical sensors (SPCEs) is highlighted in Electrochemistry, due to its advances such as low cost, sample volume reduction, miniaturization, mass production and portability [1, 2]. In addition, disposable printed electrodes overcome the problem of poisoning the surface of the working electrode due to their single-use possibility. In this work, a new self-manufactured conductive ink was developed from graphite powder as a conductive material, and a commercial glue based on polyvinyl acetate (PVA), originally used for craft purposes, as a polymeric vehicle, with the addition of solvents, acetone, ethyl acetate, and glycerin as additives. To obtain a flexible device, the substrate chosen for this purpose for the production of the sensor was the aromatic aliphatic copolyester EcoflexTM. SEM, FTIR, contact angle electrochemical impedance spectroscopy and voltammetry, carried out the morphological characterizations. The developed electrochemical sensor was applied for detection of paracetamol, using square wave voltammetry in the range from 7.0 to 100 µmol $L^{-1}$and with detection limits of 1.4 µmol L^{-1}. The sensor was suitable for rapid detection of paracetamol in artificial sweat and syrup samples with recovery between 90 and 108%. Therefore, it was possible to develop a low-cost screen-printed electrochemical sensor with satisfactory analytical response.

Acknowledgements: The authors are grateful for the financial support provided by Brazilian agencies FAPESP (2017/21097-3, 2019/24428-6, 2016/01919-6 and 2018/22214-6), CAPES (88887.504861/2020-00 and 001), CNPq (164569/2020-0, 151200/2022-0, 307070/2022-0, 301796/2022-0 and 350641/2022-6), and INEO

References:[1]R.M. Silva, A.D. da Silva, et al.,Biosensors., 13, 453(2023)[2]J.S. Stefano, L.O. Orzari, et al., Current Opinion in Electrochemistry, 32, 100893(2022)

Raman-based ultra-sensitive anti-SARS-CoV-2 antibody detection (SERS)

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During the COVID-19 pandemic, antibody-based rapid tests were democratized as the World Health Organization's main strategy to control the spreadof the SARS-CoV-2 virus and monitor its transmission. But even after pandemic control, immunochromatographic tests are still of importance in post-infection and immunological surveillance. However, regulatory agencies and healthcare managers have reported this method's poor sensitivity and reliability. Hence, improving detection methods is recommended as means to achieve high sensitivity. This work devised a technique that combines surface-enhanced Raman spectroscopy (SERS) with artificial intelligence tools to improve the accuracy, sensitivity, and reliability of the results. To do so, gold nanoparticles were synthed, characterized (UV-Vis, MET, DRX, DLS, FTIR, and Raman), and functionalized with an antigenic fragment ofthe recombinant spike protein. The spectra of 594 blood serum samples were obtained bythe Raman spectrometer Metrhom Mira XTR. Feature extraction techniques such as PCA[1], kPCA[2], t-SNE[3], and UMAP[4] were used to classify the spectra, coupled with a range of machine learning algorithms including kNN, SVM, and DT. The new system was comparedtothe standard ELISA technique and exhibited an average sensitivity of 95.8% ands pecificity of 90.8%. These findings are crucial in paving the way for a new generationof nanosensors that are highly versatile, reliable, and sensitive.

References

1. Pearson, K. On Lines and Planes of Closest Fit to Systems of Points in Space. Philosophical Magazine. 2 (11): 559-572 (1901).
2. Schölkopf, B et al. Nonlinear Component Analysis as a Kernel Eigenvalue Problem. Neural Computation. 10 (5): 1299-1319 (1998).
3. Roweis, S; Hinton, G. Stochastic neighbor embedding. Neural Information Processing Systems (2002).
4. McInnes, L et al. UMAP: Uniform Manifold Approximation and Projection for Dimension Reduction, ArXiv e-prints 1802.03426, 2018.

Acknowledgements to UFES, IFES and FAPES.

Role of oxygen functional groups in the gas sensing properties of graphene quantum dots: theoretical and experimental insights

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The monitoring of amines emitted from food degradation, such as trimethylamine (TMA), is important to ensure food quality [1]. A promising way to monitor such compounds is through the development of gas sensors, in which the choice of the sensing material is key to the sensor' s performance. Graphene quantum dots (GQDs) is a promising material for this end, due to their multiple active sites for interaction with gaseous analytes [2]. In this work, we evaluated theoretically and experimentally the adsorption of TMA onto GQDs functionalized with different oxygen functionalities, namely hydroxyl (GQDs-OH), epoxy (GQDs-Ep), and carboxyl (GQDs-COOH) groups. Density functional theory (DFT) calculations were performed with the M06-2X functional with the 6-31+G(d,p) basis set. The GQDs and the TMA structures were first optimized, and Fukui functions were calculated to support the choices of adsorptions sites. The potential energy curves (PECs) of the adsorption process were obtained based on a rigid scan calculation. Based on the well depth of the PECs, the most stable complex was formed between the TMA molecule and the edge hydroxyl of the GQDs-OH, while the less stable, was formed with the GQDs-COOH sites. To confirm the DFT predictions, gas sensing measurements were performed at room temperature by exposing the GQDs to a variable TMA concentration. The GQDs-OH sensor showed an enhanced response with sensitivity four and three times higher than the GQDs-Ep and GQDs-COOH, respectively, which is in agreement with the theoretical results. To verify the application of these sensors, GQDs-OH sensor was used to monitor the freshness of shrimp, and satisfactory results were achieved. Besides, the obtained insights can in principle be explored to develop other GQDs-based gas sensors.

Acknowledgments:
The authors thank the financial support from CNPq and Fapesp.

References:
[1] Andre, R.S et al., ACS Sensors 2022, 7, 1-28.
[2] Arunragsa, S. et al., Diam. Relat. Mater 2020, 105, 1-7

Semi-covalent imprinted polymer employed for preconcentration and determination of tetracycline in milk samples with amperometric detection in a BIA system.

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An interesting method of sample preparation is the molecularly imprinted solid phase extraction due to the selective adsorption of polymer for the target analyte. Thus, in the present work, an analytical method was developed based on the off-line preconcentration of tetracycline (TC) using a molecularly imprinted polymer and amperometric determination in batch injection analysis system using a modified screen-printed carbon electrode with AuNPs. A new tetracycline-imprinted polymer was synthesized using a semi-covalent method. In this synthesis, the template-monomer complex was synthesized by the reactionof 3-isopropenyl-α,α-dimethylbenzyl isocyanate (IPDMBI) with TC. The removal of the template by a simple thermal reaction produced specific tetracycline recognition sites. Thenon-imprinted polymer (NIP) was synthesized the same way, without the presence of thetemplate. Based on the relative selectivity coefficients, the MIP showed higher selectivity towards TC in relation to NIP, even having a smaller specific area (183.21 m2 g-1) than theNIP (206.12 m2 g-1), indicating that the greatest adsorption capacity of TC by MIP is due toimprinted effect created during synthesis. Based on the obtained results in adsorption kinetics study, the percent removal increased with time, reaching binding equilibrium in 15min for both polymers. Moreover, adsorption isotherms showed TC-rebinding capacity of
76.74 mg g-1 for IPDMBI-MIP. The method analytical presented limit of detection and quantification of 0.003 and 0.011 μmol L-1, respectively, and preconcentration factor of 214.83-fold. The applicability of the method was demonstrated by performing analysis of TC and other compounds of the family, in different milk samples, with recoveries being between 93.0-102.0%.

Acknowledgements: UEL and CNPQ.

References: [1] BARALLA, E. et al. An overview of antibiotics as emerging contaminants: Occurrence in bivalves as biomonitoring organisms. Animals, v. 11, n. 11, p. 1-17, 2021.

SERS sensing of corrosion inhibitors using Ag nanoparticles immobilized on cellulosic supports

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One of the most used methods for mitigating and controlling corrosive processes that lead to decreased flow in pipes is the use of corrosion inhibitors [1]. Both corrosion and incrustation inhibitors can be found in the water produced in oil extraction, which is a by-product of oil exploration, having a very complex matrix with the presence of numerous organic and inorganic substances, which can be very harmful to the environment. SERS spectroscopy has great potential for the analysis of corrosion inhibitors due to the great sensitivity of the technique for monitoring analytes at low concentrations, providing vibrational information of the analyzed molecules [2]. Flexible substrates constructed by immobilizing silver nanoparticles (AgNP) on paper proved to be very promising for the application of the SERS technique in complex systems. The purpose of the present work was to use paper support (cellulose) covered by a range of AgNP to build high-performance SERS substrates and to test the detection of the corrosion inhibitor benzotriazole (BZT). It was found that the power density of the laser used (λ_0= 632,8 nm) should be as low as possible to avoid degradation of the analyte. Using a nominal power of 5 mW in a Bruker Senterra spectrometer, it was possible to obtain spectra of this corrosion inhibitor with a high S/N ratio for BZT concentrations as low as $1,0\times10^{-7}$mol L^{-1}. The SERS spectra obtained are similar to those previously reported in the literature. The results indicate that it is possible to obtain SERS spectra at very low concentrations of this inhibitor on AgNP/paper substrates, which indicates the possibility of using it for more complex solutions.

Acknowledgments:
CAPES, CNPq, and FAPEMIG supported this work.

References:
[1] A. S. Fouda et al, J. Mol. Liq. 290, 111178 (2019).
[2] M. Fan, G.F.S. Andrade, A.G. Brolo, Anal. Chim. Acta 1097, 1 (2020).

Surface functionalization of magnetoelastic sensorsand gold nanoparticles for medical applications

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Magnetoelastic sensors are transducers with a working principle based on the resonance phenomenon. They are potential devices for application as biosensors in health-related areas as they respond to surface mass variations with high sensitivity. For diagnostic purposes, they are a good alternative once biological interactions cause mass increase, allowing monitoring physico-chemical parameters. However, it is necessary to functionalizethe sensor surface to promote the detection of the desired pathogen. Furthermore, whenused for detection of viral particles, it is necessary to use gold nanoparticles (AuNPs) to amplify the response signal. In this context, the objective of this work was to functionalize the surface of magnetoelastic sensors and AuNPs for application in Sars-CoV-2 diagnosis. For this, the biosensors were obtained by building biological layers of 11-mercapundecanoicacid (11-MUA) and antibodies on the sensor surface. The ATR-FTIR technique was used for characterization. AuNPs were functionalized by physical adsorption. The resonance frequency response of test (with recombinant protein for Sars-CoV-2) and control biosensors was read on a network analyzer. The ART-FTIR results confirmed the presence of 11-MUA and antibodies on the surface of the biosensors. Regarding the resonance frequency shift response, the control biosensors (in the absence of the recombinant protein) exhibited a frequency shift of approximately -40 Hz. As for the test biosensors, which were placed in contact with the recombinant protein, the frequency shift was approximately -160 Hz. The presence of AuNPs on the surface of the test biosensors was also confirmed by SEM/FEG images.

Synthesis and characterization of molybdenum-based material for the construction of sensor devices for diabetes biomarkers

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Volatile organic compounds (VOCs) can be considered non-invasive biomarkers due to the correlation that has been shown between certain VOCs and specific types of diseases through conventional breath analysis [1]. Acetone, for example, has been found to have a direct correlation with Diabetes Mellitus by analyzing its concentration in breath samples [2]. Currently, diabetes monitoring is carried out using glucose meters, which can be intrusive, painful and inconvenient [2]. Therefore, the focus of this work is to synthesize a chemical compound that is sensitive to the presence of acetone, in order to reduce the damage caused by conventional analysis methods. The material used for this purpose is based on molybdenum oxide, produced using the hydrothermal method, modified with yttrium oxide, which was added to the base material through coprecipitation. Synthesized compounds and its precursor were characterized using X-ray diffraction, transmission electron microscopy (TEM), and scanning electron microscopy (SEM) techniques. The response of the samples in the presence of acetone was monitored to verify the potential of the material for use in biomarker sensors. SEM showed that the insertion of yttrium oxide caused significant changes in the microscopic structure of the base material. Moreover, the decoration of molybdenum oxide resulted in changes in the response parameters during the analysis of electric current in the presence of acetone. Thus, this work demonstrated that the presence of yttrium oxide in molybdenum oxide causes structural changes and changes in the electric responses of the base material.

Acknowledgements:I would like to thank CAPES for the funding, Professor Orlandi for his guidance, and my research group for their support throughout the completion of this project.

References:

[1] M. Phillips, Anal Biochem, vol. 247, no. 2, pp. 272-278, 1997.

[2] Z. Wang and C. Wang, Journal of Breath Research, vol. 7, no. 3. 2013.

Synthesis and Performance Evaluation of a Graphene Oxide-Silver Nanowire Hybrid Film as a Highly Sensitive Humidity Sensor

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Humidity control is crucial for product quality and safety. For example, high humidity can spoil food and pharmaceuticals, and damage electronic devices. Reliable and accurate humidity sensors are essential to maintain product quality. In this study, a graphene oxide-silver nanowire hybrid film was synthed and evaluated as a highly sensitive humidity sensor. Graphene oxide (GO) has excellent water adsorption due to oxygenated functional groups like carbonyl, carboxyl, hydroxyl, and epoxy. Among these functional groups, hydroxyl groups (OH) have the highest potential to interact with water molecules, making GO anexcellent material for humidity sensors [1]. Silver nanowires (AgNWs) [2] and reducedhydroxyl-rich graphene oxide (rHGO) were synthed and characterized using modified polyol and Hummers methods. Humidity sensors were created by drop-casting onto interdigitated aluminum electrodes (IDE). Two types of IDE were fabricated: one with HGO only, and the second with HGO blended with silver nanowires to improve conductivity without affecting HGO's adsorption capability. The sensors were tested by placing and sealing them in pots with varying humidity levels (14% RH to 96% RH). The behavior of electric current, resistance, and impedance in response to humidity variation was observed. The sensors exhibited high sensitivity, fast response and recovery times, low hysteresis, and were compatible with real-time humidity control, such as monitoring human breathing.

[1] Q. Fatima et al., "The critical role of hydroxyl groups in water vapor sensing of graphene oxide," Nanoscale Adv., vol. 1, no. 4, pp. 1319-1330, 2019, doi: 10.1039/c8na00135a.

[2] P. Zhang et al., "Silver nanowires: Synthesis technologies, growth mechanism and multifunctional applications," Mater. Sci. Eng. B Solid-State Mater. Adv. Technol., vol. 223, pp. 1-23, 2017, doi: 10.1016/j.mseb.2017.05.002.

SYNTHESIS OF CERIUM OXIDE NANOPARTICLES OBTAINED BY MICROWAVE-ASSISTED HYDROTHERMAL METHOD

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In recent years, the search for increasing the surface-to-volume ratio of nanoparticles has demanded great efforts from the scientific community in order to understand the physicochemical properties of new nanomaterials. Increasingly, nanoparticles are developed with sophisticated molecular control in order to satisfy a wide variety of applications in various fields of science. Among the different types of materials existing in the field of nanotechnology, cerium oxide nanoparticles (NPs, nanoceria) have stood out as a promising substance in various scientific approaches, such as catalysis and biomedicine, due to its intrinsic properties, being: high thermal and chemical stability, biocompatibility and, mainly, the ability to release and store oxygen in its structure. The present study has the main objective of producing cerium (III) oxide nanoparticles, employing the microwave-assisted hydrothermal synthetic route, using aqueous cerium nitrate hexahydrate [$Ce(NO3)3.\ 6H2O$, 0.05 M] as precursor solution and potassium hydroxide (KOH, 1.2 to 6 M) as a mineralizing agent, under different radiation times at 180°C and without the addition of surfactants. The structural, morphological, and optical properties of the NPs willbe characterized by Raman spectroscopy, low-angle X-ray scattering, X-ray diffraction, field emission scanning electron microscopy (SEM-FEG), and spectroscopy in the UV-vis region, respectively. Raman spectra and XRD patterns indicate the formation of fluorite-like crystalline structures belonging to the Fm3m space group in all samples. Furthermore, it was observed that the microwave exposure time and the concentration of the mineralizing agent have a direct influence on the morphology and crystallinity of the nanostructures. By analyzing UV-vis spectroscopy measurements, maximum absorption peaks were observed between 321.29 and 335.58 nm and bandgap between 2.87 and 3.44 eV.

Synthesis of Fe2O3 nanoparticles supported on carbon nanotubes for electrochemical detection of 17-β-estradiol

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The magnetite (Fe_2O_3) and the carbon nanotubes (NTCs) are materials with excellent electrocatalytic activity, which can greatly enhance the electron transfer velocity in electrochemical sensors. [1]. This work reports the synthesis of Fe_20_3nanoparticles supported on the CNTs surface (Fe_20_3-NTCs), and their application in the construction of an electrochemical sensor for determination of 17-β-estradiol (E2). The Fe_20_3-CNTs were synthed using a hydrothermal method, where a mixture of Fe_2O_3, CNTs, $NaNO_3$, and polyvinylpyrrolidone in ultrapure water was heated at 160°C for 12 hours in an autoclave. The resulting precipitate was filtered, dried, and calcined at 600°C for 3 hours. The electrochemical sensor was constructed using an aqueous dispersion of Fe_20_3-CNTs and dihexadecylhydrogenphosphate, both in concentration 1,0 mg mL^{-1}. Then, 6.0 μL of the dispersion was dropped onto the surface of a glassy carbon electrode (GCE) and dried at room temperature for 2 hours. The Raman spectra of the Fe_20_3-CNTs, showed bands at 226 cm^{-1}(vibration mode A_{1g}), at 290 $cm^{-1}$and 406 cm^{-1}(both associated with the vibration mode $E_g$of Fe_2O_3). The energy dispersive spectra of Fe_2O_3–CNTs indicates the presence of carbon, iron and oxigen elements as expected. In the cyclic voltammograms obtained using a Fe_2O_3-NTCs/GCE is showed only a peak current, for E2, at 0,8 V vs Ag/AgCl (3,0 mol L^{-1}KCl), the current peak intensity was 1,9 times higher when compared to current response obtained with bare GCE . The electroanalytical method developed presented a good sensitivity and samples analyzed of pharmaceutical formulations, synthetic urine and lake water were analyzed.

Acknowledgements:
To the funding agencies CAPES, UEL Fundação Araucária ((243/2022-PBA) and CNPq ((307505/2021-9).

Reference:
[1] L,Ying and L, Hanbin, Electrochimica Acta 180, 1059 (2015).

Synthesis of tin(II) oxide micro-plates for carbon monoxide sensing applications

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SnO and SnO2 oxides are wide band gap oxide semiconductors with Sn+2 and Sn+4 chemical states, respectively. The first is an intrinsically p-type material while the second is n-type [1]. SnO2 is one of the most common inorganic material used in chemical sensing, especially for carbon monoxide detection. However, in the recent years, SnO has received much attention because it shows great promise as an alternative to SnO2 for use in high performance gas sensor devices [2]. The main objective of this work is the synthesis, morphological and structural characterization, and electrical evaluation of tin(II) oxide undoped and Ni-doped micro-plates for CO sensing applications. The undoped and doped (1% and 2% of Ni) tin oxide compounds were synthesized by the microwave assisted hydrothermal (MAH) method, under a reductive atmosphere. The samples were characterized using XRD, Raman spectroscopy, SEM and UV-Visible spectrophotometry. For the gas-sensing measurements, an alumina wafer was used as a substrate, with an adhesion layer of titanium and a thick platinum film used as the electrode. Films were prepared on top of the resulting substrate by a screen-printing-like method. The measurements were made in an optoelectronic device for the characterization of semiconductor materials under temperature and pressure control. The MAH method has proven to be versatile, and constitutes a viable methodology for the synthesis of SnO micro-plates under reductive atmosphere. Samples showed high chemical stability up to 400°Cand the incorporation of Ni improved sensor operation temperature. Ni-doped samples showed high conductivity at air and CO at ambient pressure, and catalized the reaction withCO, which demonstrated the potential of Ni-SnO as sensors for the detection of CO.

Synthesis of zinc tin oxide structures applied for gas sensors

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Semiconductor metal oxide materials, with ternary or quaternary type structures, have been highlighted in the scientific community due to the range of properties resulting from the type of structural alignment of the constituent elements [1,2]. This work reports the growth mechanism of zinc tin oxide-based structures synthesized by microwave-assisted hydrothermal methods and gas sensing performance for nitrogen dioxide (NO_2), hydrogen (H_2), and carbon monoxide (CO) gases. The as-synthesized materials were characterized by X-ray diffraction (XRD), scanning and transmission electron microscopy (SEM and TEM), and nitrogen adsorption/desorption experiments. Cuboctahedron-like $ZnSn(OH)_6$, "thorn-tube-like" $ZnSn(OH)_6/ZnSnO_3$, and "cactus-like" Zn_2SnO_4/SnO_2 structures were obtained by varying the synthesis time of 4h, 12h, and 24h, respectively. Gas sensor measurements showed that $ZnSnO_3$ presents an outstanding lower detection limit to NO_2, in which a 12-fold increase in electrical resistance is estimated in the presence of 1 ppb NO_2 at an operating temperature of 150 °C. Moreover, the Zn_2SnO_4/SnO_2 heterostructure exhibited superior selectivity to NO_2 relative to H_2 and CO, exhibiting a sensor response ~1500 times higher for the oxidizing gas. Hence, it is demonstrated that nanostructures growth engineering can realize ultra-selective and high-performance gas sensor devices through a greater surface area or enhanced contact potential barriers.

Acknowledgments

This work was supported by PROPe - UNESP public notice 13/2022, CNPq, CAPES, and FAPESP.

References

[1] L. Du, et. al. Inorg. Chem. Front., 6, 2311-2317, 2019.

[2] T. Bora, et. al. J. Am. Ceram. Soc., 98, 4044-4049, 2015.

Synthesis optimization of novel Zn-metal-organic frameworks based on quinolone derivatives as prototypes for adsorption of organic contaminants

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Quinolones are a class of heterocyclic organic compounds known for their classical antibacterial clinical use and other diverse biological activities. There has been an increasing interest in the use of quinolone derivatives as ligands for the construction of metal-organic frameworks (MOF) due to their unique, easily functionalized structure with agreat potential for diverse applications. In this work, a new organic ligand precursor, 1-ethyl-4-oxo-1,4-dihydroquinoline-3,6-dicarboxylic acid (IMP10), was synthesized, startingwith the condensation reaction between 4-aminobenzoic acid and ethyl ethoxymethylene malonate followed by a thermal cyclization. The intermediate obtained was submitted to anN-alkylation reaction [1] followed by alkaline hydrolysis, yielding IMP10, in its pure form. The quinolone-Zn-MOFs were initially synthesized from conditions already reported in theliterature for different systems [2]. The synthesis was accomplished by addingZn(AcO)2.2H2O into the IMP10 at ambient temperature. The reagents were dissolved in DMF and NaOH was added as a modulator. The system was incubated at 130 °C for 24 and72 h. After cooling, the separated solids were washed with DMF and EtOH, collected bycentrifugation, and dried overnight at 110 °C. The materials were labeled by their synthesisconditions: MOF-A = w/o NaOH - 24 h, MOF-B = w/o NaOH - 72 h, MOF-C = w/ NaOH - 24h, MOF-D - w/ NaOH - 72 h. The evaluation of structural parameters of these different MOFs was made by X-RD, FTIR and SEM. For further studies, the newly synthesized MOFsare going to be evaluated for the adsorption of organic pollutants in contaminated water [3].

Acknowledgements:CNPq, FAPERJ (Proc. JCNE E-26/201.416/2022), FAPESP (Proc. 2018/01258-5-2021/10885-6), and CAPES (code number 001).

References:[1] Costa, A. R. P. et al. Dyes and Pigments, 207, 110692 (2022). [2] Duan, L. N. et al. Dalton Transactions, 44, 1800 (2015). [3] Wei, F. et al. Nanomaterials, 8, 248 (2018).

Thiram and thiabendazole pesticides: effects on Agcolloid and SERS detection

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The wide use of pesticides in the scale agriculture requires the development of strategies to monitor their applications due to the great risk that they represent for the human health. The Raman spectroscopy is a vibrational technique characterized by the inelastic scattering of the light. When the target molecule is close to certain metallic surfaces, such as nanoparticles, the Raman signal can be enhanced up to 10^{10}, leading to the called surface-enhanced Raman scattering (SERS). Therefore, considering its sensitivity and selectivity (vibrational spectrum), SERS is a suitable tool for analyte detection. However, the SERS signal intensity is highly dependent on nanoparticle metal, , shape, and aggregation level, which makes its analytical application routinely a challenge for the field. Here, we have exploit two aspects of the SERS effect when applied in the detection of both pesticides thiram and thiabendazole: the effect of the pesticides on the aggregation of Ag colloids applied as SERS substrates, including the adsorption mechanism, and quantitative analysis. SERS intensity varies linearly from $1.0x10^{-8}$to $4.0x10^{-7}$mol/L with thiram concentration, leading to a limit of detection of $1.7x10^{-9}$mol/L. The loss of linearity (above 10^{-6}mol/L) was associated with changes in the Ag colloid aggregation (and morphology) due to strong interaction between the Ag nanostructures and thiram, leading to S-S cleavage with thiram adsorbing onto Ag surface through S atoms and methyl groups. In the case of thiabendazole, a linear regimen for SERS intensity was found between $1.6x10^{-7}$and $8.0x10^{-8}$mol/L, leadingto a limit of detection of ca. $7.0x10^{-8}$mol/L. Besides, the preferential TBZ adsorption mechanism onto the Ag surface was established by S atoms, leading to a sigmoidal adsorption isotherm, revealing intermolecular thiabendazole interactions and formation of Ag aggregates with distinct morphologies.

Acknowledgments:
FAPESP 2018/22214-6, CNPq, CAPES, INCT/INEO.

TUNING THE PHOTOLUMINESCENCE BY ENGINEERING SURFACE STATES/SIZE OF S, N CODOPED CARBON DOTS FOR CELLULAR IMAGINGAPPLICATIONS

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In this research, we have synthed carbon dots (CDs) co-doped with nitrogen and sulfur by facile hydrothermal method, using citric acid and cysteine as carbon source. The effect of solid-state thermic treatment (STT) at 303-453 K on the , surface, fluorescence and cellular cytotoxicity of the CDs were

systematically investigated [1, 2]. Through a simple STT, it was possible to tune surface states and the average of the CDs, causing a permanent red shift. Initially, CDs showed a decrease in cell viability with increasing concentration. However, after STT, its viability remained constant with an increase in concentration. Here, we show the possibility to label the cells cytoplasm according to the CDs fluorescence emission before (blue emission) and after STT (red emission). The CDs studied in this paper show selective luminescence properties, which are fundamental for any cell imaging application [3].

REFERÊNCIAS

[1] SONSIN, Artur F. et al. Temperature-dependence on the optical properties of chitosan carbon dots in the solid state. RSC advances, v. 11, n. 5, p. 2767-2773, 2021.

[2] RODRIGUES, Leonardo Sobreira et al. An eco-friendly green and facile synthesis of carbon dots from red propolis wax with photoluminescence dependent of reaction time and thermal treatment in solid state. Journal of Nanoparticle Research, v. 23, n. 12, p. 1-12, 2021.

[3] PUVVADA, Nagaprasad et al. Synthesis of biocompatible multicolor luminescent carbon dots for bioimaging applications. Science and technology of advanced materials, 2012.

Unlocking New Possibilities: Bacterial Cellulose-based Laser-Induced Graphene for Electrochemical Sensing Applications

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Electrochemistry and sustainable platforms like paper and bacterial cellulose offer portable, affordable, and user-friendly devices. However, current methods for creating electrochemical paper-based analytical devices (ePADs) can be cumbersome, poorly reproducible, and challenging to scale. We present a new simple, versatile, single-step technique for creating laser-induced graphene on bacterial cellulose surfaces, eliminating chemical reagents and controlled condition needs. The production process, including CO2 laser output and substrate functionalization, was optimized, and the resulting graphene was characterized, revealing a highly graphitized, porous material with a large specific surface area. The straightforward laser engraving process facilitated scalable electrode preparation and yielded outstanding reproducibility of potentiometric signals, with low variability between measurements (2.13%) and between devices (1.91%). The low-cost of the materials, the minimal equipment requirements, the single-step protocol, and the produced material's features offer a promising green, portable, and highly reproducible electrode fabrication method.

Acknowledgments
Coordenação de Aperfeiçoamento de Pessoal de Nível Superior-Brasil (CAPES)
The Brazilian National Council for Scientific and Technological Development (CNPq)

Use of an electrolyte-gated transistor to distinguish honey from molasses

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Honey is a concentrated solution of sugars, mainly glucose and fructose, produced by bees fromflower nectar or plant-sucking insect secretions. When commercialized, it is of great relevanceto know if the honey is in fact pure or may have suffered some kind of adulteration. Accordingto data from the National Health Surveillance Agency (ANVISA), approximately 30% ofcounterfeit honey is made from sugarcane molasses. This is a by-product of the sugarproduction process, where sugarcane juice is heated and concentrated to obtain crystallizedsugar. The main aspects verified in honey analysis include purity, moisture content, acidity, andsugars. Distinguishing between honey and molasses is a challenge for many people, as thesetwo products are very similar in appearance and taste. In this context, a study was conducted todevelop an electrolyte-gated transistor (EGT) and extract the typical transistors parameters toinvestigate the device capability capable of different honey and molasses. The EGT structure isindium-tin oxide (ITO) interdigitated electrodes as source and drain, poly(3-hexylthiophene-2,5diyl) (P3HT) as channel and a platinum tip for the gate. The electrolyte layer is composed ofa drop of honey or molasses. The theoretical fit was based on OFET model [1]. The used honeyis from Jataí bees ***(Tetragonisca angustula),***a stingless bee native from Brazil commonlyknown as Jataí. The extracted parameters to compare honey or molasses in the EGT were fieldeffect mobility, transconductance, on/off ratio, and threshold voltage. Preliminary resultsshowed that it is possible to differentiate honey from molasses as electrolyte in the EGT byanalyzing the parameters of the transistors.

Acknowledgments:
The authors thank CAPES, CNPq, A.B.E.L.H.A. and MCTI.

References:
[1] Horowitz, G. (2010). Interfaces in organic field-effect transistors. Organic electronics, 113-153.

Use of glass substrate functionalized with silver nanoparticles as SERS sensors for monitoring the photodegradation of organic pollutants

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Silver nanoparticles (AgNP) are widely used in environmental applications, including the treatment and monitoring of water quality. Furthermore, their favorable photophysics allows AgNP application as a sensor for the detection of different organic molecules in aqueous media through the Surface-enhanced Raman Spectroscopy (SERS) technique [1]. Therefore, the use of Raman spectroscopy combined with the properties of metallic nanoparticles, such as silver, has enabled the identification of pollutants in water, soil, and food [2]. In this scenario, we propose here the engineering of thin films composed of silver nanoparticles of different morphologies (sphere and prism) deposited onto glass slides by the blade coating method to detect the organic pollutant molecules Atrazine (pesticide), Ibuprofen (drug), and Methyl Orange (dye). UV-VIS spectra, dynamic light scattering (DLS) measurements, atomic force microscopy, and scanning electron microscopy confirmed the morphologies and of AgNP and the functionalization of the surface of the blades. The pollutant models were detected at very low concentrations (10^{-4}~10^{-6}M) in an aqueous solution by Raman spectroscopy and SERS effect due to AgNP deposition and plasmon effect. In addition, the photodegradation of these molecules was followed through the SERS effect, in which it was possible to observe changes in their spectral profile as the photobleaching occurred. These promising results augur well for further developments of AgNP plasmonic thin films for pollutants photodegradation products detection.

Acknowledgments:
CAPES and FAPESP (# 2019/01604-3) supported this work.

References:

[1] L. Mikac et al., Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 252, 119478 (2021).
[2] G.F.S. Andrade et al., Encyclopedia Of Sensors And Biosensors, 284-296 (2023).

Using Doped Carbon Quantum Dots/PVA Nanocomposite as a Platform for Nitrite Ion Detectionin Meat

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A sensor device utilizing doped-carbon quantum dots is proposed in this study for detecting nitrite in meat products through fluorescence quenching. To create the sensing platform, boron-doped carbon quantum dots functionalized with nitrogen (B,N-Cdot) were synthed through a one-step hydrothermal process using citric acid, boric acid, and branched polyethyleneimine as sources of carbon, boron, and nitrogen, respectively, resulting in an impressive quantum yield of 44.3%. Following an investigation into their chemical structure and fluorescent properties, the B,N-Cdot in aqueous suspensions exhibited high selectivity for NO_2^- in a linear range of 20 to 50 mmol L-1 under optimal conditions at a pH of 7.4 and a 340 nm excitation wavelength. Additionally, the prepared B,N-Cdots successfully detected NO_2^- in a real meat sample with a recovery rate ranging from 91.4 to 104% within the analyzed range. Consequently, a B,N-Cdot/PVA nanocomposite film was produced, which emits a blue light under excitation at 360 nm, and a preliminary detection of NO_2^- in meat products was conducted using a smartphone application. The newly developed sensing device, featuring a highly fluorescent probe, has the potential to aid in the development of a fast and cost-effective strategy for detecting NO_2^-.

UV light-assisted ozone gas sensors based on $W_{1-x}Mn_xO_3$ nanostructures

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The nanostructured $WO_3$is a metal semiconducting oxide (MOX) that has been widely used as resistive gas sensors for the detection of noxious species[1]. The photoactivation of these gas sensors is a way for its operation at room temperature of such sensor devices[1,2]. Herein, we present an investigation of the influence of annealing temperature and Mn concentration on the microstructural and surface properties of nanostructured WO_3prepared by the polymeric precursor method, in addition to the effect of Mn content on the photoactivation of the samples. X-ray diffraction analyses revealed the monoclinic structure for the $W_{1-x}Mn_xO_3$samples with an increase of crystallization degree with annealing temperature and Mn content. Scanning electron microscopy images showed that $W_{1-x}Mn_xO_3$crystals exhibit nanometric particle size. X-ray photoelectron spectroscopy analysis revealed that Mn^{+2}ions occupied the W^{+6}sites. Electrical measurements revealed that the samples were sensitive to UV and violet light stimulation. Regarding the sensing performance, experiments performed exposing the samples to different ozone (O_3) levels under UV light stimulation at room temperature, revealed that the $W_{1-x}Mn_xO_3$samples detected sub-ppb O_3levels.

Acknowledgments:The authors would like to thank the financial support provided by the Brazilian research financing institutions FAPESP, CNPq and CAPES (grant No. 88887.667095/2022-00).

References:

[1]S. R. Sriram, S. R. Parne, et al., ACS Omega 7 (51), 47796, (2022);
[2]J. V. N. de Palma, A. C. Catto, et al., Sens. Actuators Rep. 4, 100081, (2022);
[3]Z. Ca, W. Wang, et al., Sens. Actuators B Chem. 353, 131155, (2022).

ZnO presenting different morphologies: effects in the detection of toxic gases

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The detection of toxic gases is becoming increasingly important for human and environmental safety, in modern society, gases produced by fossil fuels consumption and extraction, automobiles, and factories need to be monitored to guarantee safe levels of exposure and decrease the risk of accidents [1]. Metallic oxide semiconductors (MOS) have been extensively studied for gas sensing applications since they present excellent sensitivity and response to several different gases, in addition to having excellent thermal and chemical stability and a low cost of production [1]. However, MOS exhibit a high operatingtemperature, which renders them unusable for the detection of flammable gases, and lowselectivity when exposed to more than one gas simultaneously [1]. Among metallic oxides, ZnO presents the characteristics for gas sensing and has been broadly studied, obtainable indifferent morphologies using low-cost synthesis methods [2]. In this study, ZnO was synthesized via controlled precipitation in two different morphologies named "needles" and"donuts" and after morphological and structural characterization the material was tested asa gas sensor for O_3, NO_2, CO, and acetone at different concentrations and operation temperatures to analyze the effects of morphology in the sensing properties. The sample inthe form of needles presented the best result about the detection of ozone gas while the sample in the form of donuts presented the best result concerning CO and NO_2 gases.

Acknowledgments: This study was partially funded by (CAPES) - Finance Code 001 and also was supported by FAPESP (Processes 2013/07296-2 and 2019/22076-5) and CNPq (Process 3017231/2021-6).

References:

[1] S. M. Kanan, O. M. El-Kadri, I. A. Abu-Yousef e M. C. Kanan, Sensors, v. 9, n. 10, pp. 8158-8196, 2009.

[2] L. Zhu e W. Zeng, Sensors and actuators A: Physical v. 267, pp. 242-261, 2017.

E-Advanced Materials and Surface Treatments for Biological, Dental and Medical Applications

Chitosan films loaded with different antibiotics: A comparative study on drug release

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Medical devices, such as catheters and orthopedic implants, play a crucial role in patient care. However, they can be associated with the insurgence of healthcare-associated infections (HAIs). The use of coating to prevent HAIs on medical devices is of major importance [1,2]. Recently, chitosan-based films crosslinked with tannic acid, $FeSO4$ and loaded with gentamicin [1] have shown promising antibacterial results activity for over a month. To extend the performances of this system, we investigated the impact of different antibiotics on their release kinetics and their antibacterial activity. Antibiotics with diverse chemical properties, namely, gentamicin, linezolid, trimethoprim and moxifloxacin were selected for this study. Films were synthetized with chitosan, tannic acid 20%, $FeSO4$ 5% and antibiotics 5%, then characterized in terms of thickness, which varies between 16 to 28 μm; mass loss, ranging from 15 to 30 %; and swelling, with values around 200% for all tested conditions. The antibiotics linezolid and moxifloxacin presented the highest difference in their release kinetics profiles in PBS. While linezolid was released in only three days, moxifloxacin was released over a period of 70 days with the smallest burst release. The results for the antibacterial activity corroborated the release profiles of the antibiotics over time. In conclusion, our research shows that the selection of the antibiotic significantly impacts the release kinetics from fast-acting to prolonged release. This provides valuable insights in terms of drug matrix interaction, and allows an appropriate choice of formulation for film coatings to prevent HAIs in medical implants.

Acknowledgment

BIOPARK, Prati-Donaduzzi Industry, and the Natural Science and Engineering Research Council of Canada. Thanks to Carmen and Luiz Donaduzzi, PhD, for guidance all along the project.

References

[1] Chevallier P et al.. Nanomaterials. 2023; 13:484.

[2] Wiggers et al. Front. in Bioeng and Biotech. 2022; 10:1.

DEVELOPING BIOINSPIRED MIMETIC MODELS FOR BONE PHYSIOPATHOLOGY STUDIES

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The organization of the bone tissue can be investigated from a physical, chemical, and biological point of view1. In a very simplified description, osteoblasts secrete an extracellular matrix composed mainly by type 1- collagen, in which minerals are deposited following kinetic and thermodynamic parameters. Moreover, osteoblasts also release extracellular vesicles able to regulate the concentration of phosphate and pyrophosphate (mineralization inhibitor), allowing mineralization of the extracellular matrix2. This highly controlled process can be studied in detail using biomimetic models composed by phospholipids able to reproduce in part the structure of osteoblasts′ cell membrane, collagen, and hydroxyapatite. Choosing the suitable molecules and structures to simulate the bone matrix in a reliable way is challenging. In this presentation, we will describe biomimetic models based on Langmuir films and 3D scaffolds in an attempted to reproduce the bone extracellular matrix. Lipidomics and proteomics analysis of extracellular vesicles released by osteoblast supported the choice of cholesterol, phosphatidylserine, and phosphatidylcholine and the main lipids and alkaline phosphatase and orphan phosphatase 1 as the main enzymes participating on this process. Collagen was chosen due to its abundance in the extracellular matrix. Polysaccharides were also added to the models in order to fulfil the role of non-collagenic molecules. Hydroxyapatite was chosen as a mineral model for the production of the mineralized matrices. These biomimetic models were used to study cell events related to bone mineralization and were also useful for the development of biomaterials based on surface modification of implants and membranes potentially appliable for bone replacement. Acknowledgements: FAPESP (2019/25054-2) and 408440/2021-1) and References: (1) Nogueira et al. J. Biomed. Mater. Res. 110, 967-983, 2022. (2) Bottini, M., et al. BBA Gen. Sub., 1862, 532-546, 2018.

Fatigue and Accelerated Life Testing of NanostructuredY-TZP with Non-Homogeneous Yttria Distribution

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Yttria-stabilized Polycrystalline Tetragonal Zirconia (Y-TZP) is a material with great potential for dental and medical applications due to its mechanical properties, especially its fracture toughness, however, it is prone to low-temperature degradation (LTD), which compromises its performance and survival probability. Nanometric Y-TZP with non-homogeneous yttria distribution has been shown to present improved hydrothermal degradation resistance and mechanical properties, i.e. higher fracture toughness and characteristic strength [1]. In this work, step stress accelerated life testing and the mechanical characterization of fatigued samples (mechanical cycling) were performed in a wet environment to determine a more realistic probability of survival of nanostructured Y-TZP materials with non-homogeneous yttria content. Physical (Relative density), structural (XRD and Raman Spectroscopy), microstructural (SEM and AFM), and mechanical characterizations (Accelerated fatigue testing, Biaxial flexural strength test, and Vickers hardness test) of nanostructured and conventional Y-TZP were evaluated and compared. Nanostructured Y-TZP materials with non-homogeneous yttria presented higher reliability after 50000 cycles at 800 MPa than conventional Y-TZP (83% vs. 6%) and higher characteristic strength and Weibull modulus after 2x106 cycles of 110 MPa with a frequency of 15 Hz (1419 MPa and 14.3 vs. 1157 MPa and 10.6).

Acknowledgments
The authors gratefully acknowledge the financial support of São Paulo Research Foundation (FAPESP), grants #2021/04697-2, #2021/06730-7, #2020/16500-6, #2021/06730-7 and #2022/05496-3.

References

[1] C. A. Goulart, M. M. d. Toledo Piza, T. M. B. Campos, E. A. Bonfante, and P. N. Lisboa Filho, Y-TZP nanostructures with non-homogeneous yttria distribution for improved hydrothermal degradation resistance and mechanical properties, Ceramics International, 2023, doi: 10.1016/j.ceramint.2023.04.034.

Nanostructures For Probing And Transfecting LivingCells

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Interacting with cells in a minimally invasive manner is key to performing longitudinal studies in biomedicine. One example where minimally invasive interaction with cells is important is when injecting material inside cells, either to probe cells, or to change the cell phenotype (transfection). Current delivery methods such as viral vectors electroporation and liposome delivery (lipofection) have many drawbacks. Viruses suffer from being costly, can be unsafe due to insertional mutagenesis, and are limited in cargo . Electroporation and lipofection, suffer from low cell viability and efficiency. To address this issues, nanostraws can be used to deliver cargos inside cells. Here, a review of our work using nanowires and nanostraws for developing cell transfection tools will be presented. For instance, we have used nanostraws to inject nanodiamonds in living cells. A long-standing question in the field concerns the state of the cell membrane on such hollow nanostructures. In order to investigate the state of the cell membrane during and after transfection, we have performed video-rate live cell STED microscopy imaging of the cell membrane when interfaced with nanostraws. The time-lapse STED images reveal that the cell membrane opens entirely on top of nanostraws upon application of gentle electrical pulses, and that it recovers and seals 30-60 min after turning off the electrical field. We further demonstrate that we can achieve direct delivery of fluorescent nanodiamonds to the cytosol using nanostraws. The nanostraw delivery leads to efficient and rapid nanodiamond transport into cells compared to when incubating cells in nanodiamond-containing medium. Moreover, whereas all internalized nanodiamonds delivered by incubation end-up in lysosomes, a significant larger proportion of nanostraw-injected nanodiamonds are located in the cytosol, which opens up for using nanodiamonds as cellular probes.

Treatment of titanium by electrolytic plasma, aiming application in dental implants with drug delivery system

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Pores with controlled number, size, and distribution are of fundamental importance in drug delivery systems. In dental implants, for example, they additionally need to have adequate wettability, chemical composition and biocompatibility. In the present work, titanium was oxidized by pulsed plasma electrolytic (PPEO), using 0.025 M sodium dihydrogen phosphate and 0.25 M calcium acetate as electrolyte. The samples were treated for 2 min or 6 min, using pulses of 50/100 and 100/50 (time on/off time - Ton/Toff) with a current density of 38 mA/cm2. These pulse times are shorter than those usually used in the literature. After treatment, wettability, Ca/P ratio, number, and pore distribution were evaluated. Cell adhesion/proliferation was also evaluated. It was observed that smaller and more uniform pores are obtained in smaller Ton and/or treatment time. Thus, under PPEO with pulse times close to the plasma discharge times conditions, were the most appropriate for use in implants with drug delivery systems.

Acknowledgements:CNPq- 402536/2021-5, 304422/2021-5, and 465423/2014-0 processes, and the CAPES.

Reference:

Martínez Campos E, Santos-Coquillat A, Mingo B, Arrabal R, Mohedano M, Pardo A, et al. Albumin loaded PEO coatings on Ti — Potential as drug eluting systems. Surf Coat Technol. (2015);283:44-51

3D printed scaffolds using alpha wollastonite for biomedical applications

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The 3D printing of ceramic scaffolds for bone regeneration allows for the creation of standardized structures with complex geometries and controlled porosity, making the method highly reproducible. Incorporating wollastonite, a bioactive ceramic, has shown positive results in bone formation, with low toxicity and efficient bone integration in the scaffolds. The alpha phase of wollastonite is highlighted for its thermal and mechanical stability, being more stable at lower temperatures, below 1120°C, and constituting a monoclinic structure. Additionally, wollastonite exhibits high reactivity and adequate biocompatibility, making it a promising material for applications in bone tissue engineering. The process of scaffold formation involved tests of precursor powder granulometry, alpha phase wollastonite, to determine particle size and analysis of density by helium pycnometer. 3D printing of alpha-phase wollastonite scaffolds was performed using a paste/bioink containing the precursor material and the binding agent PEG 400, to improve the mechanical properties of the scaffolds. Printing was carried out using a paste extrusion printer, followed by the sintering process and characterization of the scaffolds. Characterizations by X-ray diffraction (XRD), Raman spectroscopy, scanning electron microscopy (SEM), and rheology revealed that the scaffolds exhibited appropriate porosity and well-defined crystalline phases. As a result, the findings demonstrated that the use of ceramic scaffolds based on alpha-phase wollastonite is a promising option for biomedical applications due to their suitable mechanical and structural behavior.

Acknowledgements: This work was supported by Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP) (2022/07944-3), CAPES (88887.672990/2022-00) e Instituto Federalde Educação, ciência e tecnologia do Estado de São Paulo (IFSP SJC).

Additive manufacturing of bioinspired and functionalized microneedles with active-passive hierarchical structures for self-decontaminating action

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This work aims to produce with active materials, printable microneedles inspired by the microstructures of the stinging hairs of *Urtica dioica* (L.). To achieve this, a printable and biocompatible polymeric nanocomposite is being developed, featuring self-decontaminating action on printed surfaces. The nanometric phase of this composite was formed by silver nanoparticles reduced in a printable resin by photoreduction, aiming to confer bactericidal properties to the material [1]. Multi-walled carbon nanotubes (MWCNT) constitute another nanometric phase of the composite, used for the formation of oriented nanoneedles by applying an electric field (DC) during printing [2] to achieve self-decontaminating action against viruses on the printed surfaces [3]. MWCNT in resin was aligned by applying a constant electric field. A biocompatible resin was developed in collaboration with the Laboratory of Organic and Medicinal Synthesis (LASOM/UFPE) and used as the polymer phase of the nanocomposites in the printing of bioinspired microneedles. Cytotoxicity tests were conducted on the synthesized resins, with and without the prepared nanomaterials, in collaboration with FIOCRUZ/PE (Aggeu Magalhães Institute), which indicated the samples that had cellular viability. The printed bio-inspired microneedles were characterized by optical microscopy, which indicated that their shapes and dimensions were preserved from the model with little deviation, and their surfaces were analyzed through AFM and SEM. The synthesized resins are being characterized by FTIR and DSC.

Acknowledgments

This work was supported in part by the Coordenação de Aperfeiçoamento de Pessoal deNível Superior (CAPES) RIMA Project - Edital Capes 09/2020. and CNPq Brazilian Agencies.

References

[1] S. Sánchez-Salcedo et al., Acta Biomater. 155, 654-666 (2023).[2]
L.A. Chavez et al., J. Appl. Polym. Sci. 136(22), 47600 (2019).
[3] A.P. Mouritz et al., Ivanova, Nano Select 2, 2061-2071 (2021).

A luminescent powder for latent fingerprint development: Evaluation of the acute oral toxicity and liver histopathology of the MOF [$Eu_2(BDC)_3(H_2O)_2$]

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Abstract: The luminescent metal-organic framework (LMOF) [$Eu_2(BDC)_3(H_2O)_2$], entitled EuBDC, has been previously tested as a latent fingerprint developer, on porous, nonporous, and multicolored surfaces obtaining sharp images for comparison and identification of suspects. Since the EuBDC will be in direct contact with fingerprint experts, it is important to assess its toxicity to ensure safety in its use. Therefore, in this study we focused on the evaluation of the acute oral toxicity and the liver histopathology. The synthesis followed Reineke *et al.*[1] reported method and the material produced showed the same XRD patterns, regular europium transitions, a red-orange color when excited with UV light (254nm), with 77.4% of its particles ranging from 0.1 to 10μm. For the toxicity study, youngWistar rats were evaluated using OECD protocol no. 423. The administrated dose was closeto 2000 mg/kg and the animals were observed closely for the first 4 h, periodically duringthe first 24 h, and daily for 14 days. During this period, the following parameters were evaluated: animal behavior, water and food intake, body weight, organs macroscopic changes and weight (liver, heart, spleen, lung, and kidneys), animal mortality, and liverhistopathology. No signs of changes were observed in animal behavior, nor signs of toxicityin any of cited parameters, and in the liver tissue, when compared to the control group. Additionally, no animals died, which classified the EuBDC in the Globally HarmonizedSystem (GHS) category 5 (the least toxic), with a LD_{50}>5000 mg/kg. Finally, this LMOF canbe safely used as a latent fingerprint developer, although more toxicity studies are requiredfor a wider margin of safety.

Acknowledgements:

CNPq, CAPES and FAPDF. The University of Brasilia. Isabel Luana de Macêdo and prof. Márcio Botelho de Castro for the histological sections.

Reference:

[1] T.M. Reineke, M. Eddaoudi, M. Fehr, D. Kelley, O.M. Yaghi, J. Am. Chem. Soc. 121 (1999) 1651-1657

Atomic force microscopy used to identify polymerchains conformation

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It is already known that the relationship between microstructure and macroscopic properties in polymers is a central key to understand and control the desired results when using these materials. Ultra high molecular weight polyethylene (UHMWPE) is a polymer used as a bearing surface in orthopedic implants due to its outstanding physical and mechanical properties. Preferential crystalline orientation in its macromolecular chains can result in anisotropic physical properties. On the other hand, modifications in the structure of the polymer have a direct effect on its wear. It is because this polymer is considered a soft material and its deformation can induce transformation from amorphous phase into crystalline ones with preferred orientation [1]. Fortunately, atomic force microscopy (AFM) shows up as a powerful tool to image molecular structure of polymers. Even though it is a challenge, crystalline molecular structures can be observed with AFM operated in tapping mode [2]. In this context, the goal of this work is to image surfaces of UHMWPE subject to plastic deformation. UHMWPE subject scratch tests, under different scratching patterns, was imaged to identify crystalline orientation in the wear tracks. Besides that, UHMWPE subject to a plane strain compression was also imaged and its directional polymeric chainscould be observed. AFM images helped to understand the behavior of the macromolecularchains under both abrasive and compressive stresses.

References:

[1] L.G.Moura, E.A.Fancello, E.da Rosa, C.R.M.Roesler, C.M.Almeida and M.M.M.Moraes. J Mech Behav Biomed Mater, vol. 142, page 105816 (2023).

[2] J.KUMAKI. Polymer Journal, vol. 48, page 3 (2016).

Barium Titanate-doped Hydroxyapatite for Bone Tissue Engineering Using Freeze-Casting Process

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The present work describes the production of barium titanate modificated hydroxyapatite samples for bone tissue engineering applications using the freeze-casting process. Samples containing 0.25%, 0.50%, 0.75% and 1.00% wt% of barium titanate relative to hydroxyapatite were prepared using ball milling technique followed by freeze-casting and heat treatment. Cylindrical monoliths were evaluated using x-ray diffractometry, Fourier Transform Infrared spectroscopy, scanning electron microscopy and microtomography. Results shows peak-shifting in all difractogram and peak splitting at characteristic peak around 30.7°, as a consequence of barium titanate binding to hydroxyapatite, suggesting that barium atoms in barium titanate molecules interacted with hydroxyapatite crystals viaCa2+ ionic sites. XRD results also shows a very pure barium titanate structure, with littlebarium carbonate formation. FTIR analysis also yields characteristic hydroxyapatite band in560 cm-1, also showing characteristic bands at 478 and 598 cm-1 related to Ti-O and O-Ti-Omodes, suggesting once more barium titanate bonded to hydroxyapatite [1]. Scanning electron microscopy presents the porous structure highlighting its direction of alignment,and also showed lamellae solid structure when using dimetylsulphoxide as a solvent for freeze-casting method at -130 °C. Microtomography suggests that the porous structure wasaligned to heat gradient as expected, and also radial growth was noted to be random, due toheat conduction properties of Technyl® mold, also no sign of agglomeration or deposition ofbarium titanate was found.

AcknowledgementsFAP-DF, GRACO, University of Brasília, Federal University of Amazonas.

[1] Zarkoob, H., Ziaei-Rad, S., Fathi, M., Dadkhah, H. Advanced Engineering Materials, 14(6) (2012). Synthesis, characterization and bioactivity evaluation of porous barium titanate with nanostructured hydroxyapatite coating for biomedical application. https://doi.org/10.1002/adem.201180091

Bioprinting of Carboxymethylcellulose-Laponite Hydrogels with Dental Pulp Mesenchymal Stem Cells

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In recent years, bioprinting has emerged as a promising technique for creating functional tissues and organs by enabling the precise and controlled deposition of biomaterials and cells [1]. In this study, we investigated the printability and biocompatibility of Carboxymethylcellulose (CMC) and Laponite hydrogels using dental pulp Mesenchymal stem cells (DMSCs) and a Bio X 3D bioprinter.We formulated CMC-Laponite composite hydrogels with varying concentrations of CMC and Laponite to optimize their rheological properties, ensuring excellent printability and structural integrity. The biocompatibility of the composite hydrogels was evaluated by encapsulating DMSCs and assessing cell viability, proliferation, and differentiation potential. Our results demonstrated that the optimized CMC-Laponite hydrogels exhibited good printability, with the bioprinted constructs maintaining their shape fidelity post-printing. Furthermore, encapsulated DMSCs showed high viability and proliferation, indicating the composite hydrogel''s biocompatibility. This study paves the way for developing patient-specific bioprinted constructs that can significantly enhance the outcomes of regenerative therapies.

Acknowledgments:

The authors acknowledge the São Paulo Research Foundation (FAPESP 2017/23776-5) grant and the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq-Brazil, 308569/2018-0).

References:

[1] Deo KA, Singh KA, Peak CW, Alge DL, Gaharwar AK. Bioprinting 101: Design, Fabrication, and Evaluation of Cell-Laden 3D Bioprinted Scaffolds. Tissue Eng - Part A. 2020;26(5-6):318-338. doi:10.1089/ten.tea.2019.0298

Chitosan hydrogel containing bioactive glass aiming at3D printing for tissue engineering

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3D printing is a rising processing technique in tissue engineering due to high reproducibility and the possibility of complex or even patient-specific models. Chitosan is a promising biomaterial for both 3D printing and tissue engineering, because of its biocompatibility, rheological properties, biodegradability, and cost-effectiveness. Bioactive glasses can help the regeneration of different biological tissues and act as reinforcement for chitosan matrix. In this work, chitosan hydrogels were produced with different concentrations of 58S bioactive glass (0.5 up to 5%wt). For the rheological characterization, frequency, and recovery experiments were carried out. Printability and shape fidelity were evaluated during the 3D printing process. Indirect cytotoxicity assay using Balb/c 3T3 cells was performed to evaluate the biological properties of the scaffolds. The presence of the glass maintained the viscosity properties and increased the recovery percentage. All materials formed filaments and allowed printing with suitable form maintenance. Biological experiments showed high cell viability for all 3D scaffolds, even though a reduction was observed for higher glass concentrations. The results indicate that the studied materials are printable and potentially recommended for tissue engineering applications.

Acknowledgments:
The authors thank the financial support from FAPESP (Fundação de Amparo a Pesquisa do Estado de São Paulo, 2020/00329-6), CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico) and Capes (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior). CEM-UFABC and laboratories infrastructure from UFABC and IPEN/CETER are appreciated.

Design of bone-mimetic collagen/K-carrageenan based scaffolds for investigating bone mineralization

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In this study, we used collagen to fabricate mineralized scaffolds mimicking the microenvironment of the native bone. The sulphated polysaccharide κ-carrageenan (κ-Carr), extracted from renewable sources, was added to the scaffolds to fulfil the role of glycosaminoglycans, due to the similarity of their chemical structure, in the organization of the extracellular matrix and cell attachement.[1,2]Ordered matrices were obtained by self-assembling type I collagen molecules in highly aligned fibrils by slow evaporation of acidic solutions at high concentrations.[3]Fibrillogenesis was promoted from exposition in ammoniac vapours for 24 hours. Scanning electron microscopy images showed a surface formed by a dense and uniform network of intertwined fibrils, while from a side view, it was observed collagen fibrils ordered in a parallel alignment, which is characteristic of dense connective tissues. The incorporation of 5 wt.% κ-Carr promoted changes in the surface morphology but, the presence of collagen fibrils was still observable. After 7 days of culture, MC3T3-E1 osteoblasts were viable both in the absence and in the presence of κ-Carr. The presence ofκ-Carr in the collagen scaffolds stimulated the maturation of the cells to a mineralizing phenotype, as suggested by the overexpression of key genes related to bone mineralization, including alkaline phosphatase (Alp), bone sialoprotein (Bsp), osteocalcin (Oc), and osteopontin (Opn), as well as the ability to mineralize the extracellular matrix after 14 and 21 days. The results indicate that these scaffolds can constitute an appropriate model to study the role of the structural organization of bone-mimetic synthetic matrices in cell function. Acknowledgements: This work was supported by FAPESP (2018/25871-8). References: (1) de Wildt, B. W. M. et al. Curr. Opin Biomed Eng. 2019, 10, 107-115. (2) González Ocampo, J. I. et al. Acta Biomater 2019, 83, 425-434. (3) Giraud Guille, M. M. etal. Soft Matter 2010, 6, 4963-4967.

Development of biopolymeric composites containing bioactive glass for periodontitis treatment

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Dental caries and periodontal disease are considered the most frequent global problems that affect human oral health. However, in cases of periodontitis, the exacerbated immuneand inflammatory response of the organism itself is largely responsible for the destructionof the periodontal ligament and supporting bone. Thus, the therapeutic approach regardingthe minimization of tissue damage, as well as their recovery, can be considered as relevantas combating the disease-causing microorganisms. A promising material for this applicationis bioactive glass (bioaglass), which has been shown to be an excellent osteoinducer and efficient in inhibiting some microorganisms, but it is available in powder form, which can be lost for saliva during application. In order to restore periodontal tissues, the present work developed a film delivery system containing bioactive glass and allowing its prolonged release. Zein, a hydrophobic protein obtained from corn, was selected as the main material for film formation. When bioactive glass meets biological fluids, it forms a layer similar to hydroxyapatite, which is responsible for binding to bone. It also has osteoinductive capacity, soft tissue binding and antibacterial activity due to the high pH resulting from ion release. The results indicate that the matrix obtained is stable, and the precursors showed synergy in film formation. With the incorporation of bioactive glass, the material became even more homogeneous, with a more regular morphology and good adhesion to the mucosa. Release assays indicated that the bioactive glass exhibited ion releasing activity, releasing morethan 50% of all calcium present and therefore was active in the simulated body fluid.

Effect of surface treatment by plasma enhanced chemical vapor deposition on Ti6Al4V to improvements on anticorrosive properties focused on dental application.

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The recent increase of advanced materials and surface treatments to dental applications have motivated the exhaustive research by new materials. The electrochemical impedance spectroscopy technique is extremely useful to show the destabilization mechanism of the protective layer on Ti6Al4V alloys in the presence of a critic concentration of fluoride ions as 1500 mgL^{-1}. In this sense the protective effect of diamond-like carbon layer deposited by plasma enhanced chemical vapor deposition on Ti6Al4V was monitored by this technique. The values related to elements of simulated equivalent circuit shows better resistive properties to Ti6Al4V with the presence of diamond-like carbon layer. Bode diagrams show more than one time constant and reproduce all interlayers in contact with simulated artificial saliva (Mcllvaine buffer pH=5) containing 1500 ppm of fluoride ions. The protective behavior of the layer deposited against corrosion was evidenced by the higher values of the total impedance at lower frequencies and lower capacitance when compared to bare alloy, proving the barrier effect of this layer. The hardness (H= 20.05GPa), elastic modulus (E =172.55GPa) and ratio H/E (0.11) shows values related to high hardness and elastic modulus. The Raman spectra show Diamond e Graphite bands, at around 1377 cm^{-1}e 1557 cm^{-1}, respectively. The relation (ID/IG) at around 0.77 and width of these bands characterize this layer as a-C:H type of diamond-like carbon. It''s possible to conclude that beside these alloys showing excellent corrosion resistance due to $TiO_{(s)}$, $TiO_{2(s)}$and $Ti_2O_{3(s)}$onto alloy surfaces, the diamond-like carbon can protect this material against fluoride ions in this media.

Electrospun PVDF/HAp-β-TCP composites: Synthesis, Characterization and Biocompatibility.

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The development of new materials for tissue engineering and bone regeneration has become an increasingly important research area in recent years. Polyvinylidene fluoride (PVDF) is one of the most explored polymers for biomedical applications owing to its piezoelectric properties of its β phase. However, PVDF only does not meet all requirements, such as biocompatibility, bioactivity, and enhanced mechanical properties. In this context, PVDF has been used in combination with hydroxyapatite (HAp) and beta-tricalcium phosphate (β-TCP) to overcome these requirements. In recent decades, PVDF-based nanofibers have been successfully synthesized, resulting in a large surface contact area, which leads to enhanced properties, mainly targeting tissue engineering. PVDF/HAp-β-TCP composites were synthesized by the incorporation of HAp-β-TCP particles (with different concentrations) into the PVDF matrix solution, followed by electrospinning onto a rotating collector to produce nanofibers. Nanofiber characterizations were performed by various techniques, including X-ray diffraction (XRD), Raman spectroscopy, scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS), mechanical testing, and immersion in simulated body fluid (SBF) for different periods of time. In summary, this study aimed to investigate the synthesis and structural, microstructural, vibrational, and biological characterization of PVDF/HAp-β-TCP nanofibers produced by electrospinning. These results have relevant implications for the development of novel materials for tissue engineering and bone regeneration applications.

Enhancing photodynamic efficiency of gold shell- isolated nanorods (AuSHINRs) upon toluidine blue-o (TBO) conjugation

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Light-based therapies have shown promise in selectively destroying target cells owing to their minimally invasiveness and localized action. Among these therapeutic approaches, photodynamic (PDT) and photothermal (PTT) therapies are notable for their distinct methods of cell inactivation. PDT involves the generation of reactive oxygen species by photosensitizers, while PTT entails the localized increase in temperature induced by photothermal agents. In this work, we shall investigate the synergistic effect of combining both therapies on colorectal tumor cells (CACO2). Thus, photothermal agents based on gold shell-isolated nanorods (AuSHINRs) were conjugated with the photosensitizer toluidine blue-o (TBO), followed by a second coating with silica (AuSHINRs_TBO_SiO2) to prevent leaching. The entire process of synthesis, coating, and conjugation of AuSHINRs_TBO_SiO2 was controlled and characterized with, transmission electron microscopy (TEM), zeta potential UV-vis and fluorescence spectroscopies. The plasma membranes of the target cells were chosen as the primary focus of the photoreactions. Therefore, bioinspired cell membranes based on Langmuir and Langmuir-Schaefer monolayers were chosen to investigate the binding mechanisms and photochemical outcomes at molecular level. Furthermore, the photo-activation of AuSHINRs_TBO_SiO2 in the in vitro culture of CACO2 cells exhibited significantly higher efficiency in reducing cell viability owing to the synergy achieved. Such results demonstrated promising potential in combining both PDT and PTT, and an overview of each step in the study step will be presented herein.

Acknowledges: FAPESP (2018/22214-6) and CAPES (88887.657626/2021-00).

Evaluation of coffee by-products and rosemary extractsas natural preservatives

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Preservatives are compounds added to drug, cosmetic and food formulations to maintain their physical-chemical and microbiological stability, their efficacy and safety. Synthetic preservatives are linked to adverse effects that led consumers to use those of natural origin [1]. In this study, we used extracts of rosemary leaves and coffee by-products, using ethanol 70% as solvent, to develop a preservative system of natural origin. Coffee extracts showed 43.53 ± 3.49 mg EAG/g of sample and 2.32 ± 0.11 mg EQ/g of sample in the total phenolic tests and flavonoids, respectively, 12.5 mg/mL of minimum inhibitory concentration against Escherichia coli and 25 mg/mL against Staphylococcus aureus and Pseudomonas aeruginosa. The rosemary extract showed 134.59 ± 19.25 mg EAG/g of sample and 1.69 ± 0.13 mg EQ/g of sample in the tests of total phenolics and flavonoids, respectively, 2.5 mg/mL of minimum inhibitory concentration against E. coli, 1.25 mg/mL against P. aeruginosa, 0.3125 mg/mL against S. aureus, and 0.08 mg/mL against Candida albicans. Coffee extracts showed cell viability greater than 79%, not being considered toxic against the human keratinocyte lineage. The combination of extracts showed a partially synergistic action against S. aureus and P. aeruginosa. Thus, the extracts were incorporated into an O/W emulsion, which showed a significant change in consistency, spreadability, cohesiveness and pH parameters, compared to the formulation containing synthetic preservatives, indicating that it is a non-inert preservative system. A challenge test was also performed for the microorganisms S. aureus, E. coli and P. aeruginosa, for which the formulation met the criteria required by ANVISA. In conclusion, the association of coffee and rosemary extracts has potential to develop a new preservative system.

[1] Torfs, E., Brackman, G. (2020). A perspective on the safety of parabens as preservatives in wound care products. International Wound Journal, 18. doi:10.1111/iwj.13521

Functionalization and improvement of corrosion resistance of additively manufactured lattice structures

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Bone resorption is one of the main causes of failure in orthopedic implants. This phenomenon is caused by a mechanical mismatch between the bone and the implant, causing bone resorption and loosening the prosthesis, leading to failure [1].

The use of implants with mechanical properties that mimic those of bone may prevent this type of failure. Additive manufacturing allows the design of implants with custom-designed mechanical properties thanks to its ability to produce porous structures with complex geometries, namely lattice structures. However, these structures have some drawbacks such as their great surface area and the presence of certain zones that may favor the appearance of crevice corrosion [2].

In this work, porous lattice structures and solid bulk samples were fabricated in Ti-6Al-4V using the electron beam melting (PBF-EB) technique. The samples were functionalized using the anodizing technique in an NH_4F + $NH_4H_2PO_4$-containing electrode. The thermodynamic and kinetic parameters of the corrosion process were determined using direct and alternating current electrochemical techniques on the as-fabricated and functionalized samples. The as-fabricated lattice structure samples presented pitting attacks in phosphate buffered saline solution at 37 °C. The functionalization improved the corrosion resistance by hindering pitting attacks on the lattice structures and reducing current density.

Acknowledgments: São Paulo Research Foundation (FAPESP) - grants 2020/05612-8 and 2021/06516-5; Research supported by CTI’s Open Labs - Multiple Users and Shared Facilities, CTI Renato Archer, research institution from MCTI.

References: [1] GEETHA, M. et al. Ti based biomaterials, the ultimate choice for orthopaedic implants - A review. Progress in Materials Science, v. 54, n. 3, p. 397-425, 2009. [2] LONGHITANO, G. A. et al. Corrosion resistance improvement of additive manufactured scaffolds by anodizing. Electrochimica Acta, v. 366, p. 137423, jan. 2021.

Improving the surface functionalization of 3D printed biosensor

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The biosensor 3D-printed carbon black based on conductive filaments requires functionalization processes and geometry modification to obtain a satisfactory electrochemical response [1]. The surface microstructure of biosensor is important to control both mechanical and physical properties interface [2]. The surface generates chemical residues, and its efficiency depends on the polymeric composition of the filament. Most surface pre-treatments using chemical reagents are expenditure. Laser-Induced Graphene (LIG) activation process emerges as an attractive functionalization method, which allows simultaneously surface deformation, low hydrophobicity and better electroactivity [3]. The present work shows the electrochemical results of interaction between LIG surface of 3D-printed electrode of commercial Acrylonitrile Butadiene Styrene copolymer (ABS), called carbon black (CB), and aqueous solution. It is known that LIG promotes formation of carboxyl, epoxy and hydroxyl on the surface [3], but the oxidation of carbon depends on temperature and ABS/CB quality. Based on Nyquist plot measured data, it was observed that the LIG strategy offers direct formation of electroactive sites of functionalized surface that interacts with water and peroxide due affinity by carboxyl and hydroxyl. However, these reactions might be explained by some limitations due to porous structures presentedin its surface, to temporal electrode roughness and to non-reversible electroactivation. Weconcluded that the LIG technique for ABS/CB can be produce functionalized electrodes without controlled atmosphere and using conventional industrial process. Acknowledgements to UDESC and FAPESC. [1] G.Ye, W.Wang, D.Fan, P.He Appl. Surf. Sci. 538(1), 148117 (2021) [2] R.G.Hjort, R.A.Soares, J.Li, D.Jing, L.Hartfel, B.Chen, B.Van Belle, M.Soupir, E.Smith, E.McLamore, J.C.Claussen, C.L.Gomes Microchimica Acta 189 (2022) [3] K.H.Nam, M.Abdulhafez E.Castagnola G.N.Tomaraei, X.T.Cui M.Bedewy Carbon 188 (2022).

Influence of Interstitial Oxygen on the Structure, Mechanical Properties, and Biocompatibility of Ti-25Ta Alloys for Biomedical Applications

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Currently, technological development has contributed to an increase in the quality of life. However, with the rise in life expectancy, there is a need to construct new medical devices. A noteworthy new class of alloys is the Ti-25Ta alloy, which possesses good mechanical properties and a compact hexagonal (α') and orthorhombic (α'') structure. Ti is a highly reactive element with oxygen at high temperatures, and can naturally form a layer of titanium oxide (TiO2) on its surface. The physicochemical properties of this oxide film areimportant for evaluating the long-term stabilization of the implant and its biocompatibility. The addition of oxygen can induce material hardening, enhancing its biofunctionality, sinceO is a strong stabilizer of the α-phase. The presence of interstitial elements (O, C, N, and H)can alter the mechanical properties of materials, particularly their elastic properties. The main objective of this project is to evaluate the effect of interstitially dissolved O on the structure, microstructure, and selected mechanical properties, as well as the biocompatibility of the Ti-25%Ta alloy, with a view to future biomedical applications. To achieve these objectives, the alloys were prepared by arc melting, laminated at 1000°C, subjected to thermomechanical treatments, and doped with oxygen at three different pressures, characterized chemically, structurally, microstructurally, mechanically, and biologically.

Acknowledgments: The authors thank CNPQ/PIBIC and FAPESP (2015/50280-5) for their financial support.

References:
[1] Acharya, Srijan, et al. Materials & Design 164 (2019): 107555.
[2] Biesiekierski, Arne, et al. Acta Biomaterialia 53 (2017): 549-558.
[3] Correa et al. Journal of Alloys and Compounds 775 (2018) 158-167.

Influence of RGD motif amino acids on Cell Adhesion in nanostructured films for Cancer Diagnosis

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The Arg-Gly-Asp (RGD) motif is a short peptide sequence that recognizes integrins on cell surfaces, stimulating cell adhesion to surfaces [1]. Integrins are associated with cancer progression and metastatic potential of malignant tumors [2] and are overexpressed in circulating tumoral cell (CTCs). Techniques based on the adhesion of CTCs are being studied to develop biomaterials for promoting specific cell adhesion [3]. Immobilizing RGD in a biomaterial may be an approach to promote cancer diagnosis. This study aimed to investigate the influence of each RGD peptide on cell adhesion. Films of 3.5 bilayers were assembled by the layer-by-layer technique. Chitosan was used as polycation, and five different polyanion compositions were tested: modified alginates with arginine (R-ALG), glycine (G-ALG), aspartic acid (D-ALG), a combination of peptides (RGD-ALG) and alginate (ALG) as a blank sample. The film surface was characterized using atomic force microscopy (AFM). Assays for prostate cancer cell (PC3) adhesion were performed to evaluate the influence of peptides on the cell binding film potential. Results showed that all compositions were able to capture cells, although D-ALG depicted enhanced ability to capture cells. This fact can possibly be associated to D-ALG higher film roughness. This study provides valuable insights into the factors that influence the cell adhesion, specifically with RGD motif, highlighting the potential for developing biosensors functionalized with specific proteins.

Acknowledges: To the National Institute of Science and Technology on Photonics Applied to Cell Biology (INFABIC). To Multiuser Laboratory of the Institute of Physics (LAMULT - Unicamp). To FAPESP for the financial support (Grant n. 2020/12980-3).

References:
Hersel, U. et al. H. Biomat., 24 (24), 4385-4415 (2003).
Gribova, V. et al. Acta Biomater, 9 (5), 6468-6480 (2013).
Zhou, N. et al. Mater. Sci. Eng.: C, 118, 111477 (2021).

Interplay of degradability and protein corona formation on silica nanoparticles: implications for biomedical applications

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Degradable silica nanoparticles (NPs) have been widely recognized as promising nanoplatforms for biomedical interests due to their ability to degrade selectively, ensuring body clearance and preventing long-term retention. One strategy for degradable NPs development is incorporating redox-responsive sulfide bridges (S-S), reducible by agents overexpressed in tumor microenvironments (TME) [1]. Although the mechanism of NPs degradability is well-known, there are remaining unclear issues concerning the interactions between NPs and relevant biological media that hamper the development of nanomedicine. Blood-based fluids are rich in biomolecules (mostly proteins) that interact nonspecifically with the NPs surface, forming a so-called protein corona (PC) [2]. The presence of PCchanges the NPs chemical identity and triggers biological responses, those that also evolves concurrently with NPs degradation. To-date, the correlation between degradability and the presence of the PC has received little attention in nanomedicine studies. This work elucidated aspects that describe the link between the degradable response of NPs and their subsequent interactions with relevant biomolecules using mainly two types of synchrotron-based spectroscopies (Fourier transform micro-infrared - μ-FTIR and synchrotron radiation circular dichroism - SRCD) and imaging (transmission electron microscopy - TEM) techniques. The results obtained support the hypothesis that the interaction between blood-based components and the NPs have implications for their biodegradability and the impairment of their effective action in TME. Here, we point out the potential of using degradable NPs to advance fundamental nanomedicine research, highlighting the exploitable property of NPs' degradability for biomedical purposes.

Acknowledgements: FAPESP (processes n° 2015/25406-5 and 2021/12225-3).

References:

[1]Moghaddam et al., 9(25), 21133-21146. (2017)
[2]Galdino et al., 21(19), 8250-8257. (2021)

Mechanical properties of hydroxyapatite-zirconia composites produced by different sintering techniques

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Hydroxyapatite-zirconia (HZ) composites are promising materials for bone implants due to the combination of high bioactivity and mechanical strength that they present [1]. However, the conventional sintering (CS) process poses a challenge as the different shrinkage rates of different materials can cause micro shear and bending stresses in the composite, leading toa product with low mechanical strength [2]. To address this issue, non-conventional sintering techniques such as Microwave Sintering (MS) [1] and Flash Sintering (FS) [2] canbe employed to achieve rapid shrinkage and densification, avoiding differential shrinkagerates. In this study, we evaluated the densification, microstructure, microhardness, and toughness of HZ composites produced through three different sintering methods: CS, MS, and FS. HZ composite samples with 50wt.% hydroxyapatite/zirconia was formed into a cylindrical shape by uniaxial pressing and then sintered. CS was performed at 1200°C, for 2h, and using 10°C/min; MS was conducted at 1200°C, for 30 min, and using 100°C/min. FSwas performed with a 300 V/cm AC electric field (1000 Hz) and 20 mA/mm² electric currentdensity for 10 seconds (furnace heating rate of 20°C/min). The results showed that the HZcomposite produced by sintering techniques with high heating rates, such as MS and FS, delivers denser materials with higher mechanical properties than those produced by CS. Future studies could focus on optimizing the sintering parameters to further improve the mechanical properties of the HZ composite.

Acknowledgments:

CNPq 150523/2022-0; FAPESP 2019/14677-9; 2021/06509-9.

References:

[1] JHA, S. K.; RAJ, R. Electric Fields Obviate Constrained Sintering. **Journal of the American Ceramic Society**, v. 97, n. 10, p. 3103-3109, 2014.

[2] THUAULT, A. et al. Improvement of the hydroxyapatite mechanical properties by direct microwave sintering in single mode cavity. **Journal of the European Ceramic Society**, v. 34, n. 7, p. 1865-1871, 2014.

Nanoporous silicon microparticles in nanocellulose films: A promising approach for efficient drug delivery

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Nanocellulose (NC) is a type of cellulose composed of fibers between 3 and 100 nm in diameter. Due to its biocompatibility, high surface area, mechanical strength, and biodegradability, it is suitable for drug delivery applications [1]. On the other hand, nanoporous silicon (nPSi) consists of silicon nanocrystals embedded in an amorphous porous silica matrix. nPSi is also widely used in controlled drug delivery due to its high surface area (200-800 m2/g), biocompatibility, biodegradability, and bioresorption [2]. In this work, we embedded nPSi microparticles at concentrations ranging from 0. 1% to 1% m/v into NC film matrices to enhance control over drug release kinetics using Methylene Blue (MB) as a model drug due to its antibacterial properties. Our results showed that NC films with 1% m/m released 92% of MB in three hours, while NC films without nPSi released the same amount of MB in 30 minutes. We performed physicochemical characterizations on the samples, including FTIR, DRX, SEM, and stress strength tests. Moreover, we tested the antimicrobial activity of the films, showing their potential for biomedical applications. Finally, we developed a mathematical model based on Fick''s second law to gain insight into the MB transport mechanism into the films.

Acknowledgments
This research was funded by FONDECYT-Chile (Grant Number 11180395).

[1] HASAN, Nurhasni, et al. Recent advances of nanocellulose in drug delivery systems. Journal of pharmaceutical investigation, 2020, vol. 50, p. 553-572.
[2] FRANCA, Carla Giometti, et al. Nanoporous silicon microparticles embedded into oxidized hyaluronic acid/adipic acid dihydrazide hydrogel for enhanced controlled drug delivery. Microporous and Mesoporous Materials, 2021, vol. 310, p. 110634.

Novel wear-resistant bio-composites produced by arc-melting process: β Ti-Nb matrix reinforced with TiC, TiB or hybrid TiC/TiB

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β-type Ti-Nb alloys are considered attractive metallic materials for long-term bone implant applications [1]. However, metallic implants present poor wear resistance, and the degradation process can be intensified with the friction occurring in corrosive body fluids, such as joint prostheses [2]. Ti-based matrix composites (TMCs) represent promising alternatives for wear-resistant applications because the addition of hard ceramic particles enables to achieve the desired combination of properties. Indeed, the degree of adhesion at the matrix/reinforcement interface is the main factor for achieving the properties, and it can be designed during the composite fabrication when chemical reactions are directly involved in the manufacturing steps, defined as in-situ reactions [3]. Therefore, the present study aimed to produce novel in-situ β-type TMCs with arc-melting process. Initially, Ti-40wt.%Nb alloy was produced by arc-melting. As the second melting step, three TMCs were prepared by adding 5 vol.% of three different ceramic powders: NbC, NbB2, and B4C. The as-cast samples were investigated with optical and electronic microscopy, X-ray diffraction, Vickers hardness measurements, and tribocorrosion tests performed in a phosphate-buffered solution. The results prove the in-situ production, such as the presence of B promoted the precipitation of TiB, and C precipitated as TiC, always embedded in a β Ti-Nb matrix. Thus, the in-situ β TMCs could be produced by varying the type of reinforcement between TiB and TiC, besides the presence of both hard phases. Consequently, the β TMCs demonstrated higher tribocorrosion resistance than the unreinforced β Ti alloy.

References

[1] S. S. Sidhu, et.al, Mater. Sci. Eng. C, 111661 (2020).
[2] N. Diomidis, S. Mischler, N. S. More, and M. Roy, Acta Biomater., 8, 852 (2012).
[3] V. R. M. Gonçalves, D. R. N. Corrêa, C. R. Grandini, C. A. F. Pintão, C. R. M. Afonso, and P. N. Lisboa Filho, Mater. Chem. Phys., 301, 127597 (2023).

Ordered mesoporous silicas with different morphologies for potential applications in vaccineformulations

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Ordered mesoporous silicas (SBA-15) have been proposed as vaccine adjuvants due to their high surface area and pore volume. Besides, it is possible to modify the morphology of SBA-15 just by changing the synthesis conditions. In this study, five modifications of the synthesis conditions were performed to obtain distinct morphologies [1]. Sample 1 (S1) was synthesized by the conventional method, at 40°C and 1000 rpm. Sample 2 (S2) was synthesized under the same conditions, except for the addition of isopropanol. Samples 3 and 4 (S3 and S4) were submitted to 55°C and 35°C stirred at 300 and 500 rpm, respectively, and submitted to static conditions for 24h. Sample 5 (S5) was heated at 30°Cand stirred at 1000 rpm. Samples were characterized by SAXS, SEM, TGA, DSC, NAI, andFTIR and showed an ordered mesoporous structure with similar lattice parameter values.NAI analyses pointed out that the SBA-15 samples have a specific surface area variation from 933.4 (S1) to 587.1 m2g-1 (S4) and pore volume variation from 1.2 (S1) to 0.7 cm3g-1(S4), and similar pore size for all samples. SEM analyses confirmed that different morphologies were obtained, such as rope-shaped aggregated rods (S1), nanospheres (S2), filiform rods (S3), hexagons (S4) and platelets (S5). Through the TG/DSC and FTIR analysis, it was verified that the mesopores are free and with their primordial groups of silanols exposed for the functionalization and incorporation of biomolecules. Thus, it can be concluded that it was possible to modify the morphology of SBA-15 without significantly altering the structural and textural properties, such as, S1 and S2 samples that have distinct morphologies but similar specific surface area, pore volume and pore size distribution. These results qualify these materials for incorporation with vaccine antigens and in vivo immunogenic assays.

Acknowledgements:

FAPESP 17/17844-8, 19/08582-5, 22/08360-5, 19/19567-7.

References:

[1]Lee, H. I. et. al. J. Mater. Chem., 8483 (2010).

Phthalocyanine-loaded ureasil-polyether materials associated with photodynamic therapy for vulvovaginal candidiasis

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Vulvovaginal candidiasis affects about 75% of women, caused by *Candida albicans*. Antimicrobial Photodynamic Therapy is an alternative treatment strategy. Hexadecafluorophthalocyanine is a phthalocyanine (PC) with a potent ability to generate reactive oxygen species (ROS) compared to other PC. Thus ureasil-polyether (UPEO) materials are capable of incorporating hydrophobic molecules, allowing their use in controlled drug delivery systems [1]. The objective of this work was to develop UPEO containing PC against *C. albicans*. The UPEO was obtained from a reaction of modified alkoxide and modified polymers. The infrared spectrum (FTIR) was performed under scanning in the range of 4000-650 cm-1. In vitro evaluation of the antifungal activity was analyzed by the agar diffusion method. The UPEO and UPEO-PC, did not show cracks or precipitates, and the UPEO-PC showed intense blue, the UPEO proved a macroscopic uniformity with a diameter of 1.6 cm and a thickness of 0.2 cm. The spectrum of FITR showed the characteristic bands of PC and UPEO, we highlight the band at 1130 cm-1 referring to the C-N bond of the pyrrole rings and UPEO keeps its bands at 1083 cm-1 (C-O or C-N), and 1642 cm-1 (C=O). In TG, two mass loss events, the first close to 100 °C with 3.12%, related to volatile elements, and the second event between 273 and 510 °C relatedto 67% of mass loss, related to the decomposition of the carbonic structure. The antifungal activity demonstrated that UPEO-PC presented better activity (7 mm inhibition) compared to free PC (without inhibition), this response is relating its solubility increase. The results pointed out that when incorporated into UPEO the solubility is improved as well as the antifungal activity.

Acknowledgments:
UEPB, FAPESQ, CNPq and CAPES.

References:

[1] SANTILLI, Celso Valentim et al. Controlled drug release from ureasil- polyether hybrid materials. Chemistry of Materials, v. 21, n. 3, p. 463-467, 2009.

Phthalocyanine-nanostructured lipid carrier dispersedin hybrid materials associated with photodynamic therapy against *Candida albicans*

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Candida albicans causes diseases such as vulvovaginal candidiasis (VVC) affecting ~80% of women at least once in their lives. Antimicrobial photodynamic therapy (aPDT) is based on oxidative photochemical reactions. Hexadecafluorophthalocyanine is a phthalocyanine (PC) that needs a shorter pre-irradiation time to achieve the ideal CFU for clinical use. Nanostructured lipid carriers (NLC) are second-generation lipidic nanoparticles. Hybrid materials (UPEO) maintain the physicochemical properties of dispersed materials [1]. This work aimed to produce NLC containing PC dispersed in UPEO associated with aPDT for VVC. The melt-emulsification method resulted in the carriers without instability events for 90 days. The UPEO obtained by the reaction of modified, alkoxide and polymer, were geometrically equal and the absence of fissures or bubbles. FTIR spectra showed no deletion of clusters and no emergence of new bands indicating that the chemical structures are preserved. The TG showed an improvement in the stability of NLC when dispersed in UPEO, in ~100 ºC the NLC-PC had a mass loss of 85%, when dispersed in the UPEO they presented about 3%. DTA revealed two endothermic events to NLC-PC around 100 ºC referring to the loss of solvents or dehydration. The binary mixture of PC-UPEO or NLC-PC revealed no changes in peak temperature or enthalpy. In the microbiological test by agar diffusion method, with *C. albicans*, the isolated PC did not present an inhibition halo, in the no-light group, the PC-UPEO group showed a 4 mm. In the group with light, AM and PC-UPEO, NLC-PC, and NLC-PC-UPEO showed halos of 10 and 7, 10 and 15 mm, respectively, showing an improvement in the solubility and activity of PC. The formulation produced has the potential to be the treatment for VVC caused by *C. albicans*.

Acknowledgments:
UEPB, FAPESQ, CNPq and CAPES.

References:
[1] ROSATI, Diletta et al. Recurrent vulvovaginal candidiasis: an immunological perspective. Microorganisms, v. 8, n. 2, p. 144, 2020.

Physico-chemical characterization of six commercial chitosan and impact on antibacterial releasing

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Chitosan is a biocompatible and biodegradable biopolymer that has high potential to be used as coatings for medical devices to prevent Healthcare-Associated Infections. Despiteits many advantages, there are drawbacks when translating the coatings production frombench to industrial scale. The most notable is the variable quality of raw material, whichbrings difficulties to standardize and ensure consistent quality of finished biomedical products. In this study, six chitosan from different suppliers were characterized and used tosynthetize films with gentamicin according to a previously developed protocol [1]. The aimwas to determine the impact of raw chitosan characteristics, such as molecular weight (Mw)and deacetylation degree (DDA) and purity on film properties and antibiotic release kinetics. Chitosan Mw varied from 110 to 340 kDa, DDA from 82 to 94%, proteins content from 0.14 to 0.49 %, moisture from 7 to 12%, and ash content from 0.6 to 1.5%. This high variability resulted in different films properties, such as thickness, ranging from 20 to 22μm, mass lossin PBS, from 21 to 30% and swelling behavior in PBS, with values varying from 104 to207%. All the synthetized films showed antibacterial activity against S. aureus and E. coli atleast up to 30 days. However, the films exhibited different antibiotic release profiles, ranging from 42 to over 70 days of antibacterial activity. In conclusion, chitosan sourcingimpacts the antibiotic release kinetics on the tested films. Therefore, the characterization ofthe raw chitosan, Mw, DDA and purity, is crucial in determining the final film properties,affecting the final application in terms of short or prolonged release time.

Acknowledgement

BIOPARK, Prati-Donaduzzi Pharmaceutical Industry, and the Natural Science and Engineering Research Council of Canada. Thanks to Carmen and Luiz Donaduzzi, PhD for their help and guidance all along the project.

Reference

[1] Chevallier P et al.. Nanomaterials. 2023; 13(3):484.

PLA/Strontium-substituted hydroxyapatite scaffolds for Bone Tissue Engineering

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The development of new materials for bone tissue engineering is a promising solution to the issue of autogenous replacement. For so, fractures in the bone can be healed using the approach of tissue engineering, which proposes structures to support tissue growing (scaffolds), signaling molecules, and cells combined [1]. The strontium-substituted hydroxyapatite (HASr) is a promising ceramic material used to improve the properties of poly(lactic acid) (PLA) scaffolds [2]. In this work, scaffolds were produced by Fused Filament Fabrication, a simple 3D printing technique, with PLA/HASr filaments obtained using a single-screw extruder. Afterwards scaffolds were treated with NaOH solution (0.01M) at 50°C for 15 min. A Two-Level Fractional Factorial Designed of Experiments wasapplied for the processing parameters in the extruder (2^{5-2}): the three zones temperatures,pull roller speed, and screw speed. The diameter and distribution of ceramic in filamentswere the response variables. The results point out the great effect of Zone 3 and screw speed in response variables. This data was used to produce filaments with 1,76±0,06 mm ofdiameter, with good dispersion of hydroxyapatite, evaluated by SEM. The scaffolds had adimensional accuracy of up to 13.98±1.85 %. Thermal analyses (TGA/DTA) indicate that theaddition of HASr provides thermal stability for the scaffolds, and SEM images show theformation of micropores on the surface of the material. In vitro tests indicated that PLA/HASr scaffolds presented no cytotoxicity.

Acknowledgments: CBPF for gently providing the hydroxyapatite. This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de NívelSuperior - Brasil (CAPES) -Finance Code 001 and by the FAPERJ.

References:

[1] M. Maia-Pinto et al., polymers v. 13(74), 1 (2021)

[2] L. Scudeller et al., Materials Science and Engineering C vol.79, 802 (2017)

PMMA-silica coatings modified with calcium phosphates and silver particles for bioactive protectionof Ti6Al4V alloy

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Surface properties play a key role in osseointegration and the long-term performance of implants. In this context, surface modification with coatings has shown to be a promising route to inhibit the release of toxic metal ions and provide bioactive and antibacterial properties [1]. Therefore, this study focuses on the corrosion protection of Ti6Al4V implants using PMMA-silica coatings modified in the near-surface with calcium phosphates (CaPs) and silver (Ag). The hybrids were prepared by the sol-gel process combining the hydrolytic-condensation reactions of tetraethylorthosilicate (TEOS) with the radical polymerization of methyl methacrylate (MMA) and 3-methacryloxypropyl trimethoxysilane (MPTS). CaPs powders of hydroxyapatite (HA) and β-tricalcium phosphate (β-TCP), and Ag nanoparticles were added into the sol-gel phase prepared from the acidic catalysis of TEOS and MPTS. The 12 μm-thick bi-layer coatings are homogeneous, defect-free, and exhibit strong adhesion to the substrate (>12 MPa). The structural properties of the hybrids were evaluated using infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermal analysis, and contact angle measurements. FTIR and XRD results confirmed the formation of the PMMA-silica phase as well as the presence of crystalline CaPs and Ag particles. The higher surface hydrophilicity of coatings (~65°) containing CaPs and Ag compared to bare Ti6Al4V alloy (77°) favors the contact with the tissues. The additives do not affect the structural integrityas evidenced by the long-term stability and high corrosion resistance of the coatings in simulated body fluid (SBF). Electrochemical impedance spectroscopy results of coatings in contact with SBF solution showed low-frequency impedance modulus values of up to 32 GΩ cm^2, remaining for 240 days more than 3 orders of magnitude higher than bare Ti6Al4V.

Acknowledgments:
This work was supported by CNPq, CAPES, and FAPESP.

References:

[1] Raphel, J., et. al Biomaterials, 84, 301-314, 2016.

Polymeric microPLates for sustained drug delivery: Atop-down low-pressure fabrication approach

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Poly(D,L-lactide-co-glycolide) (PLGA) microparticles are a versatile drug delivery platform with several applications in the pharmaceutical field [1]. Our group has developed PLGA-microPLates (µPL) with a square base of 20×20 µm and a 10 µm height. µPL was designedto deliver a broad range of therapeutic payloads, including anti-inflammatory and anti- cancer drugs, small molecules for neurodevelopmental disorders, and RNA nanoparticles for intracellular deposition [2-3]. Herein, the morphological and pharmacological properties of curcumin-loaded PLGA µPL were optimized by introducing new steps in the original fabrication protocol. Specifically, 20×20×10 µm wells, realized by soft lithography in a PVA sacrificial template, were carefully filled with PLGA and curcumin dissolved in acetonitrile. Vacuum suction was introduced to control the solvent removal and also, two different 'pre-purification' steps were tested. Four µPL configurations were realized using PLGA inputs ranging from 2 to 10 mg per preparation. The resulting µPL were characterized by SEM, Confocal, Fluorescence Microscopy, Multir Counter and pharmacological properties were performed via HPLC analyses. Depending on the initial PLGA input, the fabrication yielding ranged between 10 and 70%, the encapsulation efficiency varied from 2 to 13%, and the 1-month cumulative release ranged between 60 and 95%. Higher fabrication yielding and drug encapsulation, and slower release were associated with lower PLGA inputs. In conclusion, a systematic analysis conducted on CURC-loaded PLGA µPL allowed the authors to optimize the fabrication protocol and identify PLGA inputs to enhance morphological and pharmacological properties.

Acknowledgment: Marie Skłodowska-Curie Grant 754490 and RISE-2019 "MEPHOS" Grant 872648.

References:

[1] Vlachopoulos, A, et al. Pharmaceutic 2022, 14, 359.

[2] Di Francesco M, et al. J Control Release; 2020; 319:201-212.

[3] Bellotti E, et al. Drug Deliv Transl Re 2022; 12(8):1829-1842.

Preparation of PVP/bentonite/AgNP nanocompositefilms

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In view of the broad biological application of silver nanoparticles, the production of a lamellar silicate polymer nanocomposite, constitutes an interesting material for study. Thus, the objective of this work is to synthesize and characterize the film composed of the PVP/Bt/AgNP nanocomposite using pvp as a reducing and stabilizing agent for nanosilver. The colloidal suspension was characterized by UV-Vis DLS and zeta potential and FAAS. The thin film was formed and characterized by UV-Vis spectrophotometry. After that, bentonite was added in different proportions and interference in film formation was verified. The film was characterized using DRX, FTIR, SEM, EDS and mechanical tensile tests. The colloidal suspension was obtained and showed stability and a slow silver reduction aspect by PVP. The thin film obtained revealed the non-interference of the drying process in the synthesis of nanoparticles. The films produced showed, through XRD, that there was formation of the PSL nanocomposite. The FTIR indicated the participation of groups containing oxygen and nitrogen, derived from PVP, in the reduction of silver. The surface morphology of the samples was altered with the addition of bentonite and nanosilver. The composition was confirmed by EDS without showing contamination peaks in the material. By mechanical test, the addition of bentonite improved the initial properties conferred by PVP. Thus, it is legitimate to infer that there was formation of a film with adequate properties for biological application.

Shining light on photosensitizers incorporated in lipid membranes

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The photodynamic therapy (PDT) has been a promising alternative for treating different carcinomas. However, the lack of detailed knowledge on the mechanisms of action prevents exploitation of the therapy full potential. This work is focused on the photosensitization of lipid membranes and underlying mechanisms of the photodynamic efficiency in the treatment of cancer cells. Specifically, Langmuir and Langmuir-Blodgett (LB) films will mimic the cell membrane of tumor cells, where photosensitizers are further incorporated and subsequently photo-activated. The goal is to investigate the effects of lipid oxidation onthe physicochemical properties of the membrane, which include surface area increase and cleavage of lipid chains. In addition to the traditional surface pressure measurements (π), vibrational spectroscopy techniques are applied to the Langmuir (PM-IRRAS) and LB (FTIR) films in order to elucidate the reaction mechanisms at the molecular-level. The final goal is to correlate the findings at the molecular scale with photodynamic efficiency in complex systems, involving the in vitro culture of cancer cells. The efficiency of the photosensitizers will be quantified using flow cytometry, which will also allow for the determination of the cell death processes that are triggered, including apoptosis, late apoptosis, or necrosis. In summary, this work shines light on the molecular mechanisms of cell membrane photo-oxidation, with broad implications for PDT.

Synthesis and characterization of magnetic MCM-41 applied as drug delivery system

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Drug immobilization and delivery systems based on magnetic materials – mainly, iron oxides – have received increasing interest in the last years. The magnetic property adds to the system functionalities such as the possibility to be magnetically carried/separated, to use magnetic energy for heating (hyperthermia treatment) and to use iron as a contrast agent to diagnose (theranostic agents). To provide surface area for drug encapsulation mesoporous ordered silica, MCM-41, can be used because silica is a stable biocompatible material. In this work, a magnetic MCM-41 was synthesized using a modified synthesis with magnetic particles dispersed in the reaction medium. The material was characterized by X-Ray diffraction, Mossbauer spectroscopy, magnetism measurements, N2 adsorption-desorption isotherms and transmission electron microscopy (TEM) images. The X-ray diffractogram in high angles (15 - 80º) and Mossbauer spectroscopy show patterns corresponding to the magnetite and maghemite phases. While in the low angles (1 - 4º) of X-ray diffractogram itis possible to observe the characteristic peaks of hexagonal ordered silica (interplanar distance is about 3 nm) in a large form due to the presence of iron oxide decreasing the long-range ordering. Magnetism measurement show saturation of 10 emu g-1. The N2 adsorption-desorption isotherm is compatible with ordered silica. The obtained surface area (BET method) value is 630 m2g-1 and the pore size distribution (BJH method) shows maximum curve point in 2.7 nm. In TEM images is possible to see the ordered pore structures with larger magnetic particles (about 120 nm of diameter) dispersed in silica. Preliminary drug (chloroquine) encapsulation tests using incipient wetness impregnation were evaluated by fluorescence spectroscopy and it indicates a promising drug delivery system.

Synthesis and evaluation of poly(propylene fumarate)-grafted graphene oxide as nanofiller for porous scaffolds

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Recent advances in polymer chemistry enabled the development of advanced scaffolds for tissue engineering. A variety of materials including hydrogels and porous nanocomposites have been investigated as candidates for bone repair[1]. In an effort to obtain porous scaffolds with improved mechanical properties and biocompatibility, the current study discusses nanocomposite materials based on poly(propylene fumarate)/N-vinyl pyrrolidone (PPF/NVP) networks reinforced with polymer-modified graphene oxide (GO@PPF). The GO@PPF nanofiller was synthesized through a facile and convenient surface esterification reaction, and the successfully functionalization was demonstrated by complementary techniques. The PPF/NVP/GO@PPF porous scaffolds obtained using NaCl as porogen werecharacterized in terms of morphology, mechanical properties, sol fraction, and in vitro degradability. SEM and nanoCT examinations of NaCl-leached samples revealed networks ofinterconnected pores, uniform in size and shape. We show that the incorporation of GO@PPF in the polymer matrix leads to a significant enhancement in mechanical properties, as suggested by a decreased sol fraction for the scaffolds containing a higheramount of GO@PPF. Moreover, the surface of mineralized PPF/NVP/GO@PPG scaffolds isuniformly covered in hydroxyapatite-like crystals having a morphology and Ca/P ratio similar to bone tissue. Furthermore, the preliminary biocompatibility assessment revealed a good interaction between PPF/PVP/GO@PPF scaffolds and murine pre-osteoblasts in termsof cell viability and proliferation.

Acknowledgment:This work has been funded bya grant of the Romanian National Authority for Scientifc Research and Innovation, CNCS - UEFISCDI, project number PN-III-P1-1.1-TE-2021-1107, TE 110/2022

Toxicity evaluation of α"-$Fe_{16}N_2$ nanoparticles

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In this study, the main objective was to synthesize iron nitride nanostructures through pulsed laser ablation of a metallic iron target submerged in liquid nitrogen using a Nd:YAG laser [1,2], and to test the nanotoxicity of the samples. Morphological and structural analyses based on transmission electron microscopy (TEM) revealed the formation of monophasic α"-$Fe_{16}N_2$ nanostructures. The produced nanoparticles remained stable for several months in saline solution without any evidence of iron oxides, demonstrating the effectiveness of this method in obtaining a highly complex iron nitride phase [2]. Characterization of the nanoparticles using UV-Vis absorbance and Raman spectroscopy techniques corroborated the TEM analysis, indicating the formation of the iron nitride phase. Due to the reported high saturation magnetization and strong magnetic anisotropy of α"-$Fe_{16}N_2$ nanostructures, suspensions of α"-$Fe_{16}N_2$ nanoparticles in saline solution were tested for biocompatibility in biomedical applications [3]. The nematode ***Caenorhabditis elegans*** was employed as an alternative animal model for toxicity testing. The results indicated that α"-$Fe_{16}N_2$ nanoparticles did not induce toxicity in the nematode, although a reduction in reproductive behavior was observed [3]. This low toxicity suggests a high potential for the use of α"-$Fe_{16}N_2$ nanoparticles as drug carriers and in the development of biosensors for medical treatments and alternative therapies, such as magnetic hyperthermia.

[1] GUBERT, G. at al. Journal of Alloys and Compounds, v. 725, p. 519-525, 2017, DOI: 10.1016/j.jallcom.2017.07.176.
[2] GUBERT, G. at al. Journal of Alloys and Compounds, v. 856, p. 157392, 2021, DOI:10.1016/j.jallcom.2020.157392.
[3] GUBERT, G. at al. Nanotoxicology, v. 4, p. 1-12, 2022, DOI: 10.1080/17435390.2022.2099768.

Upconversion Nanoparticle@Metal–Organic Framework (UCNP@MOF) core-shell nanostructures as theranostic materials for treatment and imaging of cancer

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Cancer treatment remains a significant challenge in the medical field, and the use of innovative materials such as Metal-Organic Frameworks (MOFs) as drug carrier and upconversion nanoparticles (UCNPs) as bioimaging agent, have shown promise in the development of theranostic systems. This study aimed to develop a core-shell material using NaYF4:Yb3+, Er3+ upconversion nanoparticles (UCNP) modified with polyacrylic acid (PAA) and ZIF-8 to evaluate their potential for the controlled antitumor drug release. Physicochemical characterizations revealed that the structures of MOF and UCNP remained unchanged after core-shell formation. Besides that, the luminescence spectra obtained with the excitation at 980 nm by the Yb3+ and Er3+ ions showed that the material has emission in the green and red regions, respectively. The particles of the UCNP-PAA@ZIF-8 material showed an average size of 75 nm, with a 20 nm-thick ZIF-8 shell. Besides that, the porous nanosystem presented encapsulation efficiencies of 57.5% and 68.0 % for methotrexate and doxorubicin drugs, respectively. The release rate was higher in acidic medium (Phosphate-Buffered Saline, PBS, pH 5.0) than neutral medium (PBS pH 7.4) suggesting that the nanocarriers are promising for targeted drug delivery and tumor microenvironment-responsive drug release. In vitro cell viability assays, determined by MTT assay, in MRC-5 and A-549 cell lines with a higher concentration (0.1 mg. mL-1) and incubated for 48 h, showed a cell viability higher than 70 % that confirms the potential of the UCNP@MOF systems as theranostic materials. In conclusion, the multifunctional materials prepared in this work allow the potential application of these core-shell UCNP@MOF superstructures in both treatment and imaging modalities of cancer diagnosis.

Ureasil-polyether hybrid materials as a novel miconazole controlled-release system for the treatment of vulvovaginal candidiasis

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Miconazole (MCZ) is an antifungal drug used for the treatment of vulvovaginal candidiasis (VCC). The available formulations of MCZ are unable to control its release and require a high frequency of administration. Ureasil-polyether hybrids are good candidates for controlled-release drug delivery systems and can be molded into different pharmaceutical forms [1]. This study aimed to develop ureasil-poly(ethylene oxide) (U-PEO) and ureasil-poly(propylene oxide) (U-PPO) ovules loaded with MCZ for the treatment of VCC. The hybrids were obtained by the sol-gel method, loaded with 0.135 mg of MCZ, and molded as ovules. Physicochemical characterization was performed by Fourier transform infrared spectroscopy (FTIR), thermogravimetry (TG), and differential thermal analysis (DTA). The activity against ***C. albicans*** ATCC 10231 was analyzed by broth microdilution. The drug release assay was performed in a USP apparatus 2. FTIR and DTA curves did not indicate chemical or physical incompatibilities between MCZ and the tested hybrids. TG curves showed increased thermal stability of MCZ in U-PEO and U-PPO, with shifts above 50 ºC in the main thermal event of the drug. MCZ showed activity at the minimum inhibitory concentration of 2.5 μg/mL. Different release profiles were observed for the hybrids, while more than 50% of MCZ was released from U-PEO in the first 24 hours, about 7% of the drug was released from U-PPO in the same period, reaching ~ 90% and 25% of release within 120 hours, respectively. Thus, our study demonstrated a novel controlled-release system for MCZ based on ureasil-polyether hybrids with the potential to be administered at a lower frequency than the available formulations for VCC.

Acknowledgments:

UEPB, FAPESQ, CNPq and CAPES.

References:

[1] J.A. Oshiro Junior, M. Paiva Abuçafy, E. Berbel Manaia, B. Lallo da Silva, B.G. Chiari-Andréo, L. Aparecida Chiavacci, Drug delivery systems obtained from silica based organic-inorganic hybrids, Polymers. 8(4) (2016) 91.

Ureasil-polyether hybrid vaginal ovules for co-administration of curcumin and miconazole against *Candida albicans*

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Curcumin (CUR) and miconazole (MCZ) are molecules with activity against ***Candida albicans***. The synergism between these drugs improves clinical efficacy. Ureasil-polyether hybrids can be used for dual drug delivery in a single pharmaceutical form [1]. This work aimed to develop ureasil-poly(ethylene oxide) (U-PEO) ovules loaded with CUR and MCZ for the treatment of vulvovaginal candidiasis (VCC). U-PEO was loaded with 0.135 mg of CUR and MCZ, and molded as ovules. Physicochemical characterization was performed by thermogravimetry (TG), differential thermal analysis (DTA) and Fourier transform infrared spectroscopy (FTIR). The activity against ***C. albicans***ATCC 10231 was evaluated by broth microdilution. The synergism between the drugs was analyzed by the checkerboard method. The drug release assay was performed with a USP dissolution apparatus 2. TG results showed high thermal stability of CUR and MCZ in U-PEO (>260 ºC). DTA curves did not suggest physical incompatibilities between the drugs and U-PEO. FITR results indicated no chemical incompatibility between the drugs. CUR and MCZ showed minimum inhibitory concentrations (MIC) of 256 and 2.5 μg/mL, respectively. The synergistic effect was proven and MIC values were reduced to 32 and 0.625 μg/mL after the drug combination. In U-PEO/CUR and U-PEO/CUR/MCZ ovules, the release profile for CUR after 24 hours was 48% and 38%, reaching a plateau after 72 hours with 90% and 66%, showing that MCZ influenced the release profile. Our results showed for the first time a controlled-release pharmaceutical form for co-administration of CUR and MCZ as a novel combination therapy for the long-term treatment of VCC.

Acknowledgments:
UEPB, FAPESQ, CNPq and CAPES.

References:

[1] B.B. Caravieri, N.A. de Jesus, L.K. de Oliveira, M.D. Araújo, G.P. Andrade, E.F. Molina, Ureasil organic-inorganic hybrid as a potential carrier for combined delivery of anti-inflammatory and anticancer drugs, ACS Applied Bio Materials. 2(5) (2019) 1875-1883.

3D Cryo(Bio)printing Smart Oxygen-generating Bioinks reinforced with micro and nano-fibers for Tissue Engineering Applications

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To date the application of soft hydrogels to bone and cartilage regeneration is limited due to low mechanical modules (fracture and stiffness). To solve this, cryo(bio)printing surges as an alternative to improve the crystallinity of cross-linked hydrogels. At same time, the lack of oxygenated bioprinted hydrogels construction can generate hypoxia inside hydrogels and promotes cellular apoptosis. Oxygen supply for in vitro cell culture and in vivo 3D bioprinted structures is challenges to improve vascularization inside hydrogels structures. Although some technologies employ oxygen-generating materials to add sufficient oxygen levels, the limitations and challenges of current technologies include the lack of controlled, sustained, and prolonged release of the oxygen. Moreover, the fabrication methods may leave some impurities or residues resulting in toxicity to the cells and low vascularization inside hydrogels constructions. "Click" chemistry is a facile, versatile, and compatible chemical strategy to engineer hydrogels for tissue engineering applications. Herein, we disclose the engineering of oxygen-generating micro/nanoparticles in cells-laden bioinks through a combination of gelatin-methacryloyl (GelMA) and a versatile catalyst-free tetrazine and norbornene inverse electron demand diels-alder click reaction hydrogels. Different layers containing or non-micro/nanofibers were also evaluated looking for improve of mechanical properties. The hydrogels combined GelMA, chondroitin sulfate (CS) and poly(ethylene glycol) (PEG) crosslinked in situ, displaying tunable rheological and mechanical properties after nanofibers mats incorporation (special after three layers), for sustained and prolonged oxygen-release. All biological assays will be discussed and compared to other hypoxic hydrogels constructions reinforced with nanofibers mats.

3D printed Al_2O_3 scaffolds coated with calciumphosphates enriched with strontium

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Additive manufacturing is being extensively explored in the fabrication of scaffolds for tissue engineering. However, the of the pores obtained is still a challenge. In this sense, this work aims to obtain scaffolds from alumina filaments (Al_2O_3) coated with calcium phosphates (CaPs) enriched with strontium ions (Sr^{2+}) using the biomimetic method. The scaffolds were chemically treated to improve the adhesion of CaPs layer during biomimetic coating. Then, one part was coated with synthetic body fluid (SBF) 5x more concentrated and another part was coated with the same solution enriched with 1.00 mmol/L Sr^{2+}. The scaffolds were characterized by X-ray diffraction, scanning electron microscopy, and X-ray computed microtomography. Furthermore, the release of Sr^{2+}in the SBF solution was studied. It was possible to observe the formation of phosphate phases of biological interest such as α-TCP, β-TCP, HAp, TTCP, and Sr-Hap on the surface of the coated scaffolds. Regarding the morphology of the scaffolds, high porosity and high interconnectivity between pores were observed, with dimensions favorable to osteoconduction, osteoblast growth and vascularization of the tissue. The results obtained show promising properties for targeted applications of these scaffolds in the field of bone tissue engineering.

Acknowledgments:

The authors gratefully acknowledge the financial support of CAPES (Coordination for the Improvement of Higher Education Personnel).

A drug-drug solid dispersion of mefenamic acid with pyrazinamide

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There are several strategies to improve the solubility and dissolution rate of drugs when these factors are limiting drug bioavailability. Solid dispersions (SD) are used to improve these physicochemical properties of drugs. Mefenamic acid (MFA) is a non-steroidal anti-inflammatory and pyrazinamide (PZA) is an anti-tuberculosis drug. It belongs to classes II and III of the biopharmaceutical classification system, respectively [1, 2]. This work aimed to obtain a new drug-drug solid dispersion of MFA with PZA by the solvent evaporation method. The samples were characterized by X-Ray diffraction (XRD), thermogravimetry (TG), differential thermal analysis (DTA) and differential scanning calorimetry (DSC). The data obtained by XRD evidenced the formation of a SD from the interaction of MFA with PZA in molar ratio 1:1. The TG curve of this material showed good thermal stability up to 132.7 °C (Tonset). The DTA curve showed an endothermic event characteristic of sublimation of PZA at 167.4 °C (Tonset), followed by phase transition, fusion and decomposition of MFA at 183.3, 229.8 and 244.8 °C (Tonset), respectively. The DSC curve presented an endothermic of sublimation of PZA present in SD at 168.1 °C (Tonset), followed by phase transition and fusion of MFA at 183.1 and 230.1 °C (Tonset), respectively. The DTA and DSC curves did not show the phase transition of PZA in SD, indicating the interaction between starting compounds. Thus, this new drug-drug solid dispersion is a promising material to produce more effective drugs for the treatment of inflammations, pain and tuberculosis.

Adsorption of Arginine on Cellulose Nanofibers: Implications for Biomedical Applications

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Effective immobilization of biomolecules is a crucial technology to give materials appropriate functions for biomedical applications. Arginine (Arg) is an amino acid with healing properties. Its incorporation into polymers can lead to innovative materials to treat wounds. Here, we evaluated the adsorption of arginine to TEMPO-oxidized cellulose nanofibers (o-CNF) by force spectroscopy by atomic force microscopy (AFM-FS) and quartz crystal microbalance with dissipation monitoring (QCM-D). The conversion of hydroxyl to carboxylate groups on the o-CNF surface prompts a high surface density of negative charges to promote arginine absorption through the guanidinium group at pH 7. To analyze the electrostatic interaction, spherical AFM probes were functionalized with o-CNF with two degrees of oxidation (160 and 1160 μmol of carboxylate groups per g of cellulose) and their interaction with mica substrate in an aqueous medium was measured before and after the incubation in an Arg solution (0.1% m/m). During the tip approaching, the repulsion between the mica surface and o-CNFs tips becomes evident at approximately 200 nm and reaches a maximum between 15 and 35 nm. For lower (higher) charged nanofibers, a force of 0.10 nN and 0.04 nN (0.23 nN and 0.11 nN) was measured before and after arginine adsorption, corresponding to a decrease of 60% (52%) in the repulsion force and confirming the interaction between -COO_ and guanidinium groups. Furthermore, the repulsive profile of the force-distance curves after ARG incubation suggests that the ARG molecules shield not all carboxylates. In situ adsorption of Arg onto o-CNFs was analyzed by QCM-D. The mass increment of the o-CNF sensor was measured after 10 minutes of an Arg solution (0.1% m/m) flow, preliminary results indicate that the ARG adsorbs on o-CNF. This study suggests that the incorporation of Arg molecules into o-CNFs has the potential to create materials with sustained release of ARG to promote healing.

Adsorption of chlorhexidine by highly charged synthetic mica (Na-mica-4): a possible antibacterialmaterial

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Microbial resistance has become a worldwide concern due to the possible human and animal health complications these microorganisms can cause [1]. Controlled drug release systems have been carried out as an alternative, mainly using clay minerals as the delivery vehicle [2]. Thus, this work aims to study the formation of new hybrids based on chlorhexidine and highly charged synthetic mica (Na-mica-4). The materials were characterized by XRD, their adsorption potential of mica with chlorhexidine at different concentrations was analyzed, and the final material was applied in an antibacterial assay against bacteria against ***Staphylococcus aureus***ATCC 25923 (Gram-positive) and ***Escherichia coli***ATCC 25922 (Gram-negative). The XRD standards for Na-mica-4 and hybrids with chlorhexidine did not show significant differences in the reflection peaks, mainly those corresponding to the (001) plane, with values at 2θ = 7.30º (1.2 nm), indicating that chlorhexidine interacts on the outer surface of the clay mineral. The maximum amount of incorporated/adsorbed chlorhexidine was 552 mg g^{-1}, indicating good interaction between the species, with the best fit being the Freundlich model, with an R2 of 0.97. Thus, in the results of antibacterial activity, it was observed that the chlorhexidine/mica material obtained an inhibition rate of 100% against S. aureus and E. coli, thus indicating a possible new antibacterial material.

Acknowledgments: The authors thank CAPES, CNPq, and FAPEPI.

References

[1] Wu S.C., Liu F., K. Zhu, Shen J.Z. (2019). Natural Products That Target Virulence Factors in Antibiotic-Resistant Staphylococcus aureus. J Agric Food Chem. 67, 13195-13211.

[2] de Oliveira L.H., de Lima I.S., dos Santos A.N., Trigueiro P., Barreto H.M., Cecília J.A., Osajima J.A., da Silva-Filho E.C., Fonseca M.G. (2023). Monitoring the antimicrobial activity of bentonite-chlorhexidine hybrid. Mater Today Commun. 34, 105352.

Advances in electrochemical modification of Nb surface for applicability in metallic biomaterials

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Nb2O5 is a promissing ally for implantable biomaterials and eletro-optical applications, pizeoeletric materials fibers, catalysis and biossensor. Low cost techniques should be considered for Nb2O5 production. In this review, different conditions of eletrochemical anodization of pure niobium (Nb) will be considered for the formation of Nb2O5, under constant voltage applied to the surface of Nb, a growing pit forms over time and with different electrolyte types, resulting in the Nb2O5 layer. The results will be discussed based on ethylene glycol + NH4F+ H2O 4%, 60min, voltage range from 2 to 20V, temperature of 50 ±0,5°C, in vivo cell adhesion tests compared to potential voltage limits of 200 to 250V, for 2M OOCH3, 0.1MH3PO4, and 2M EDTA solution, galvanostatic and galvanostatic/potenciostatic regimes, 60s, 300s or 30 min, current density of 350mA/cm2, and post-anodization chemical etching using 9M H2O2 and 0.3M HCl at 75 °C for 30min. The formation of the Nb2O5 oxide layer is observed under all electrochemical conditions, with fracture occurring only at 250W. Post anodization chemical corrosion, allowed significant in vitro cell adhesion and the addition of nano-roughness to the micro-pures of the surface. Cell proliferation was by confirmed in vivo. Further studies are suggested to investigate chemical corrosion on the oxide surface.

Acknowledgements

This work has been supported by the following Brazilian research agencie CAPES Finance Code 001 and FINEP.

References

[1] P. Canepa et al., “A Two-Step Approach to Tune the Micro and Nanoscale Morphology of Porous Niobium Oxide to Promote Osteointegration”, Materials, vol. 15, no 2, p. 473, jan. 2022, doi: 10.3390/ma15020473.

Antibacterial nanostructured coatings on high touch surfaces to pre-vent pathogen transmission in hospital settings.

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A major worldwide challenge is the prevention of Health Care Associated Infections (HCAI). Environmental screening confirms that pathogenic microorganisms can persist for a long time, contaminating items, equipment and "common" area in hospital causing se-rious infections and epidemic outbreaks [1]. To mitigate public health risk, the main strat-egies include the disinfectants' usage and high Touch Surfaces (HTS)' cleaning which could be regarded as a critical vehicles for bacteria via hands [2]. However, these methods ap-pear ineffective to inhibit bacterial growth and often detergents use may lead to envi-ronmental consequences, as well as increasing the risk of the onset of resistances. This ev-idence justifies the increased interest in the identification of new approaches based on antimicrobial coatings on sanitary surfaces [3], likely made of nanomaterials, to reduce microbiological contamination.
The objectives of this study are: i) the development and characterization of new antimi-crobial nanostructured removable polymeric coatings (AMC) for healthcare setting sur-faces, based on chitosan and polycaprolactone formulations; ii) the evaluation of in vitro efficacy of the innovative AMC, iii) the preliminary on site validation of the developed AMC, by its application on HTS (i.e. bed side table or bars) in hospital rooms.
Results show that the developed AC samples are effective in reducing bacterial growth, even in common indoor environment conditions. Furthermore, in vitro results show that a rapid bactericidal action obtained by using the new developed nanoparticles is a very important aspect for the correct identification of the active coatings to be used.
Acknowledgment
The project NANOBIOSAN, funded by INAIL BRIC ID49, is kindly acknowledged.
References:
1. A. Eichner et al., Journal of Hospital Infection, V104, I1, P85-91 (2020)
2. B. Brooks et al., Nat. Commun, V8, P1814 (2017)
3. S. Chyderiotis et al., C. Microbio

Applied natural hydrogel functionality

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Tissue engineering is recommended as a promising therapy for tissue repair and can be effectively used in nerve, bone and skin tissues (DONG; MA; GUO, 2020). Polylactic acid (PLA) is a polymer with good biocompatibility that can be used in bone tissue engineering. Chitosan, in turn, has antimicrobial activity and is indicated for use in biomaterials. Methacryloyl gelatin is also a versatile biomaterial with tunable properties and compatibility for use in tissue engineering (HUANG et al., 2021). This work aims to research the state of the art in order to verify publications (patents and articles) consolidated in the literature on the functionality of applied hydrogels. The technological prospection was carried out in three databases: ESPACENET, USPTO and WIPO. These databases were selected due to their accessibility and reliability of available data. The search for journals was carried out on the main platforms, namely: Web of Science TM, Scopus and Scielo. Data collection was performed in February 2023, using descriptors, keywords and terms: "suture", "hydrogel", "chitosan" and "poly(l-lactic acid)". large number of deposits in the years 2015 and 2020 inthe EPO base, both with 23 patents deposited. China is the most advanced and developed country in hydrogel and chitosan sutures, with a total of 54 patents deposited, followed by the United States with 43, the base of WIPO with 42 and Canada with 24 deposits, of which countries and organization with greater quantity and progress when it comes to this area.

REFERENCES:

DONG, R.; MA, P. X.; GUO, B. Conductive biomaterials for muscle tissue engineering. Biomaterials, v. 229, 2020.

HUANG, C. et al. Effect of kartogenin-loaded gelatin methacryloyl hydrogel scaffold with bone marrow stimulation for enthesis healing in rotator cuff repair. Journal of Shoulder and Elbow Surgery, v. 30, n. 3, p. 544-553, 2021.

Assessing the Photodynamic Efficiency of Rose Bengal Against Colorectal Cancer Cells

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Colorectal cancer is currently the third most prevalent cancer type worldwide and is responsible for the second-highest number of cancer-related deaths. To address this issue, novel therapies are being explored to efficiently destroy cancer cells while minimizing adverse side effects. Photodynamic therapy (PDT) is a non-invasive therapeutic modality in which a photosensitizer (PS) is applied to trigger cancer cell death through photochemical mechanisms. Rose Bengal (RB) is an amphiphilic PS from the xanthene family that exhibits high quantum yield in singlet oxygen ($^1O_2$) generation, which is a reactive specie that can oxidize biological structures. Herein, we evaluated the effect of RB on cell membranes of colorectal carcinoma (Caco-2) by assembling Langmuir monolayers on concentrations ranging from 1 to 25 × 10^{-6}mol/L. The photodynamic efficiency of RB was also investigatedat different concentrations (0.5 to 25 × 10^{-6}mol/L) and incubation periods (0.5, 3, and 24hours) on in vitro culture of Caco-2 cells. The viability of cells was assessed by MTT assays24 h after 1-h irradiation at 525 nm (32.3 mW/cm2) in in vitro experiments. The results ofLangmuir films indicated that RB interacts with membrane lipids, leading to their photooxidation upon irradiation and suggesting a possible damage to the cell membrane. MTT assays demonstrated that concentrations above 5 × 10^{-6}mol/L reduced the cell viabilityby 80%, irrespective of the incubation period. In summary, RB has shown potential as a photosensitizer in the photodynamic therapy of colorectal cancer.

Acknowledgments: FAPESP (18/14692-5, 18/22214-6, 20/15854-9, 21/14020-0, 22/02189-2) and CNPq for financial support.

Bioactive three-dimensional scaffolds inspired by bone extracellular matrix

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Carrageenan (Carr), a group of natural sulfated polysaccharides extracted from red algae of the Rhodophyceae family, has been widely applied in tissue engineering [1]. Its use is justified by the similar chemical structure compared to glycosaminoglycans, a matrix of polysaccharides present in the bone extracellular matrix [2]. Carr is capable of forming thermoreversible gels and its gelling behavior depends on the molecular characteristics of each Carr group, i.e. the amount of sulphate groups, and the addition of electrolytes [3]. Thus, the objective of this study was to synthesize and characterize 3D biomaterials containing Carr with improved properties to stimulate the biomineralization of osteogenic cells. In the first part of this study we analyzed the mechanical properties of hydrogels and membranes containing 3 and 5 wt % of κ- and ι-Carr with the addition of KCl, NaCl, and CaCl2 as crosslinkers. With the aid of a texturometer, we evaluated the mechanical behavior of the hydrogel and the stress, strain, and Young''s modulus of the membranes. The results showed that κ-Carr forms stronger materials when compared to ι-Carr. Even more, higher concentrations of Carr results in higher strength and resistance values. Also, each electrolyte acts differently on the groups of Carr, depending on the formulations as hydrogels or membranes. As next steps, we intend to evaluate the osteogenic properties of these samples in 3D osteoblasts cultures and their respective bioactivity and biodegradability. Summing up, we will correlate how different structures of Carr-based biomaterials affect the mechanical properties and the osteogenic, bioactive and, biodegradable properties.

Acknowledgments: Capes (88887.718965/2022-00), FAPESP (2020/08787-0).

References: [1] Nogueira, et al., ACS Biomacromolecules, 24(3), 1258-1266, (2023); [2] Yegappan, R. et al., Carbohydrate polymers, 198, 385-400, (2018); [3] Thành, T. T. et al., Macromolecular chemistry and physics, 203(1), 15-23, (2002).

Biological evaluation of gelatin biomaterial viewingskin tissue engineering

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In some adverse scenarios of the human organism, the skin healing process might not happen naturally. As a consequence, the wounds originated can evolve to the chronic stage, where there is a deficiency of the natural production of collagen [1]. For that reason, the biomedical industry has a need to seek alternatives that can stimulate skin regeneration, such as biomaterials, biomolecules and cellular therapies based on tissue engineering. Biomaterials based on hydrolyzed collagen, also known as gelatin, are commonly used for tissue engineering studies because of its biocompatibility and biodisponibility, moreover, it can be used as a scaffold for cell culture [2]. Thus, this project had the objective to carry out cytotoxicity assay with the gelatin biomaterial, during a period of up to 2 days with Vero cells culture in a 12-well plate. Following the formation of the cell monolayer, fragments of the gelatin were incubated to the culture and, after 48 h, the inverted light microscopy was held to analyze the cell morphology, characterizing the qualitative cytotoxicity of the biomaterial. The microscopy analysis did not show any kind of morphological changes in the cells, which had the standard elongated shape with the presence of round nucleus and nucleoli. Hence, the experiment executed during the development of this study evinced that hydrolyzed collagen is more beneficial for the skin tissue engineering area, because of its cost, the favorable properties and the cell's response to it. Going further, other studies can be performed viewing the combination of biomolecules, e.g. growth factors, with the tridimensional structure of the gelatin biomaterial.

Acknowledgements:

I thank the Tissue Engineering and Biomolecules UFABC’s research Group for their support and UFABC for the undergraduate scholarship.

References:

[1] Vig K and Chaudhar IA, et al. Int J Mol Sci, vol.18, page 789 (2017).

[2] Barbanti SH, et al. Polímeros: Ciência e Tecnologia, vol. 15, pages (2005).

Biomaterials based on calcium polyphosphate, alginate, and silver nanoparticles as candidates for antibacterial and bone applications

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Biomaterials in health sciences are becoming more constant to treat bone-related problems caused by chronic joint diseases in the elderly, diseases and deformities that affect growth in childhood, traumas caused by traffic accidents, and others.[1] For these biomedical applications, bone substitutes and grafts require a set of properties, such as biocompatibility, angiogenic, and osteoinduction potential, biological safety, adequatemechanic properties, and high stability.[2] In this context, calcium polyphosphate (CPP) emerges as an alternative to develop bone biomaterials because it has biocompatibility, low toxicity, osteoconductive, osteoinductive, and osteogenic potential, as well aspresents controlled porosity. Despise those characteristics, the properties of CPP-based medical devices can be enhanced, by adding other additives, such as polymers (e.g.,alginate) and nanoparticles (e.g., silver nanoparticles - AgNPs), to improve their mechanical and antibacterial properties, respectively.[3] Therefore, this work pretended to develop CPP-based materials for bone applications, incorporating alginate and AgNPs.The results showed that AgNPs presented spheric morphology and antibacterial potential against E. coli and S. aureus. Diffractometry results revealed that composites have crystalline phases of hydroxyapatite, dicalcium phosphate, and tricalcium phosphate. The biomaterials based on CPP, alginate, and AgNPs were characterized by FTIR and SEM, confirming the formation of the composites, which have the potential to be used as antibacterial biomaterials for bone applications.

Acknowledgements:The authors thank the UFPE, CNPq, FACEPE (IBPG-1324-1.06/21) and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020).

References:

[1] BAROLI, B. Journal of Pharmaceutical sciences, v. 98, n. 4, p. 1317-1375, 2009.
[2] HAUGEN, V.; et al. J Clin Periodontal, v. 46, n. 21, p. 92-102, 2019.
[3] MONTE, J.P.; et al. Materials Letters, v. 338, 2023, 134027

Buriti oil in obtaining polyurethanes: a new approachto biocompatible materials

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Recent studies have investigated the potential of buriti for developing healing formulations due to its ability to accelerate the tissue regeneration process and reduce inflammation as oil or polymer. Among these, polyurethanes are a class of polymers that find applications in industrial areas and medicine due to their excellent properties. However, the synthesis of polyurethanes usually requires the use of toxic and non-renewable petrochemical materials, leading to environmental concerns [1]. Therefore, researchers have been exploring sustainable alternatives for the synthesis of polyurethanes [2]. In this context, the work proposes the use of buriti oil in the manufacture of biocompatible polyurethanes. The monoacylglycerol (MAG) was obtained by reacting buriti oil and glycerol, in the molar ratio 1:2, in the presence of the catalyst LiOH and confirmed by FTIR. Polyurethanes were synthesized from MAG in the presence of hexamethylene diisocyanate (HDI) and polyethylene glycol (PEG).Acknowledgements: FAPEPI, CAPES, UFPI, LIMAV.References: [1] Pacheco, I. K. C. ; Reis, F. S. ; Carvalho, C. E. S. ; Matos, J. M. E. ; Argolo Neto, N. M. ; Baeta, S. A. F. ; Silva, K. R. ; Dantas, H. V. ; Sousa, F. B. ; Fialho, A. C. V. . Development of castor polyurethane scaffold (Ricinus communis L.) and its effect with stem cells for bone repair in an osteoporosis model. Biomedical Materials, v. 16, p. 065006, (2021). [2] Da Silva, Francisco I.; SÁ, Égil; Matos, José M.E. Polyurethanes Obtained by Reacting 1,6-HDI with Monoglycerides from Babassu Oil: A DFT Study. Journal of the Brazilian Chemical Society, vol. 31, p. 1949-1954, (2020).

Calcium phosphate ceramic with TiO2 nanoparticles: mechanical and chemical characterization

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Purpose/Aim: Develop a sustainable bovine hydroxyapatite dental ceramic with the addition of titanium dioxide (TiO2) nanoparticles (5% and 8% by weight), and analyze the outcome of this addition to the microstructure, mechanical and chemical properties, according to the International Organization for Standardization (ISO) 6872:2015 for dental ceramics.. Materials and Methods: Disks (Ø = 14 ± 2 mm; thickness = 1.2 ± 0.2 mm) were obtained through uniaxial and isostatic pressing from bovine hydroxyapatite powder and TiO2 nanoparticles and sintered at 1300ºC for 2h. Three experimental groups were developed (HA, HA+5%TiO2 and HA+8%TiO2) and microstructurally characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). For mechanical and chemical analysis, indentation fracture toughness (IF), biaxial flexural strength (BFS) and chemical solubility tests were performed. Results: XRD spectra revealed the appearance of a peak corresponding to b-tricalcium phosphate (ß-TCP) for HA group. For HA+5%TiO2 and HA+8%TiO2, the entire composition was converted into ß-TCP and calcium titanate (CaTiO3). The SEM images showed a dense ceramic matrix and a uniform distribution of another phase in groups with TiO2 nanoparticles. IF toughness was higher for HA+5%TiO2 (1.34 ± 0.26 MPa.m1/2) and HA+8%TiO2 (1.28 ± 0.21 MPa.m1/2) than for HA (0.65 ± 0.10 MPa.m1/2). HA showed significantly higher characteristic stress (295.8 MPa) in comparison to groups with 5% (235.1 MPa) and 8% (214.4 MPa) TiO2 nanoparticles. Differences were not observed between the Weibull modulus values. The solubility results indicated that all experimental ceramics were above the 2000 ug/cm2 limit set by the ISO 6872:2015. Conclusions: The experimental calcium phosphate ceramic with additions of 5% and 8% of TiO2 achieved desirable mechanical properties, but the chemical solubility values did not allow it to become a dental ceramic.

Characterization of alterations in the lipid fraction of microencapsules enriched with açaí lipid extract

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The açaí (Euterpe oleracea) presents itself as a berry fruit widely consumed and with antioxidant capacity related to its lipid composition. Lipid antioxidant substances are highly labile, which requires research around drying techniques such as spray-dryer and freeze-drying in order to increase shelf life and minimize the decomposition of compounds. The microencapsulates were formulated from the pulp of frozen natural açaí from a local supplier. For preparation, defrosting was performed under refrigeration and filtration toseparate the aqueous extract from the gummy portion. The gum was used to promote solvent extraction under maceration, to obtain the lipid fraction (control group). The extractwas subjected to spray-drying, stufa with air circulation and freeze-drying. Of the microencapsulated, lipid fraction and composition study were obtained, being el and s: ultraviolet scanning of the samples, gas chromatography, high performance liquid chromatography, DPPH and TBARS antioxidant assays, FTIR and thermal assays. Among the findings, it was possible to identify, in açaí oil, a composition rich in unsaturated fatty acids,as well as tocopherols and carotenoids, in addition to evidencing that drying techniques act directly on this composition, preserving it, in the case of lyophilization, and degrading it, in the case of drying in an oven and by spray-dryer, due to hyperthermic exposure , being evidenced, although the exposure to UV light caused greater degradation of lipid fractions, culminating in the identification of degradation products and reactive oxygen species. In terms of antioxidants, açaí oil showed high activity (dose-dependent) and the TBARS assayalso showed that lipid oxidation was more evident in samples submitted to heating, with greater formation of malonaldehyde, highlighting the benefits of freeze-drying in this vegetable oil.

Characterization of bioactive glass/$Co_XFe_{3-X}O_4$ superparamagnetic nanocomposites and their potential application in bone cancer treatment by magnetic hyperthermia

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Bioactive glasses have been applied in bone tissue repair and regeneration due to their biocompatibility and osteogenic properties. If these glasses are designed to display magnetic properties, they can also be applied in bone cancer treatment by magnetic hyperthermia, thereby coupling bone regeneration and cancer therapy. This work aimed at synthesizing nanocomposites consisting of cobalt ferrite nanoparticles ($Co_XFe_{3-X}O_4$, X = 0.15, 0.12, and 0.06) dispersed in a matrix of a sol-gel-derived bioactive glasses based on the $58SiO_2$-$33CaO$-$9P_2O_5$system. The structural and magnetic properties of the nanocomposites were characterized by scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS), X-ray diffraction (XRD), and vibrating sample magnetometry (VSM-SQUID). The scanning electron microscopy and X-ray diffraction results confirmed that the nanoparticles are embedded in a nanoscale glass matrix, thus forming a nanocomposite. Magnetic characterizations evidence their superparamagnetic, highlighting exchange interactions among the magnetic nanoparticles within the nanocomposite. Calorimetric measurements of the nanocomposites showed a proper heating behavior for magnetic hyperthermia. The developed superparamagnetic bioactive nanocomposite display tuned magnetic properties compared with other similar materials published in the literature, which are addressed to cobalt doping. Then, they are promising materials for applications in bone cancer treatment allied with bone regeneration.

Acknowledgments:

The authors thank the financial support from FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo, 2020/00329-6), CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico, 141760/2022-2) and Capes (Coordenação de Aperfeiçoamento dePessoal de Nível Superior, cod 001). CEM-UFABC and laboratories infrastructure fromUFABC and UNICAMP are appreciated.

Characterization of TiO_2 nanotubes and electrochemical resistance behavior of the anodizedTi-35Nb-5Ta alloy

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Titanium alloys that contain non-toxic elements such as Ta, Mo, Nb and Si are interesting for biomedical applications, as they combine high corrosion resistance, biocompatibility and mechanical resistance. The lifetime of an implant also depends on its surface properties, which can be improved by growing TiO_2 nanotubes through the anodizing process [1]. In this study, Ti-35Nb-5Ta alloys were electrochemically anodized in order to evaluate the formation of nanostructured layers and the corrosion resistance of the alloys. Surface characterization was performed by scanning electron microscopy (SEM) coupled to an energy dispersive spectroscopy (EDS) probe, X-ray diffraction (XRD) and atomic force microscopy (AFM). Electrochemical characterization through polarization curves and electrochemical impedance spectroscopy in simulated body fluid (SBF) [2]. The results indicated that it is possible to grow TiO_2 nanotubes from the parameters used in the anodizing process. Nanotubes were observed throughout the surface of the samples, in addition to good uniformity of the layer and good regularity of the diameter of the nanotubes. The anodized samples showed high resistance to corrosion, due to high values of polarization resistance and corrosion potential, in addition to low values of corrosion and passivation current densities. Thus, anodized Ti-35Nb-5Ta showed sufficient corrosion resistance to be used in implants.

Acknowledgements:

FAPEMA

References:

[1] CHEN, Q; THOUAS, G.A. Metallic implant biomaterials. Mater. Sci. Eng. R Rep, vol. 87, p. 1-57, 2015. 2015;87:1-57.

[2] NOGUEIRA, R.P. et al. Characterization of Optimized TiO2 Nanotubes Morphology for Medical Implants: Biological Activity and Corrosion Resistance. International Journal of Nanomedicine, v.16, p. 667-682, 2021.

Combining Photothermal (PTT) and Photodynamic (PDT) Therapies for Enhanced Therapeutic Efficiency on Skin Cancer Cells

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Cancer is one of the leading causes of mortality worldwide. Skin cancer, which accounted for 19.3 million cases in 2020, constitutes one-third of all diagnosed cancers. In the quest for minimally invasive clinical treatments, light-based therapeutic approaches such as photothermal (PTT) and photodynamic (PDT) therapies have been explored. Metallic nanoparticles act as photothermal agents, converting light into heat, leading to cell death through hyperthermia. Meanwhile, photosensitizers (PS) generate reactive oxygen species (ROS) upon light stimulation, triggering cell death through oxidative stress. The synergistic combination of PTT and PDT has been proposed as a strategy to enhance therapeutic efficacy by leveraging their dual effects. In this study, gold nanoparticles were coated with silica (AuSHINs), conjugated with the PS methylene violet 3RAX (VM), followed by a second silica layer, resulting in the nanostructure named AuSHINs_VM_SiO_2. UV-Vis spectroscopy confirmed the maximum absorption bands of both VM and AuSHINs in the green region, at 555 nm and 527 nm, respectively, enabling the simultaneous activation of PTT and PDT. AuSHINs with VM were verified by DLS, zeta potential, TEM and Raman measurements. From these experiments, the monodispersity were confirmed, resulting in the synthesis of nanoparticles with a diameter of 13 nm and, after coating and conjugation, 24.45 nm. Subsidiary experiments of temperature increase and generation of singlet oxygen ($^1O_2$) were performed for AuSHINs and AuSHINs_VM_SiO_2 by irradiation at 525 nm. Thus, they showed a temperature increase of about 14°C and the VM demonstrated efficiency as a PS agent ($\Phi_\Delta {}^1O_2 = 0.157$). The therapeutic efficiency of these nanostructures will be evaluated on in vitro cell cultures derived from primary (A375) and metastatic (SH-4) melanoma compared to PDT and PTT by MTT assays.

Acknowledgements: CNPq and FAPESP.

Comparative study of the electrochemical behavior ofas-cast and heat treated Ti-12Mo-13Nb alloys and Ti-based alloys commercially available

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Titanium alloys are widely used for biomedical applications due to their excellent mechanical properties, biocompatibility, and corrosion resistance. However, the presence of alloying elements such as vanadium (V) and aluminum (Al) can negatively affect their biocompatibility [1]. Therefore, there is a growing demand for titanium alloys without V and Al for use as biomaterials. These alloys must have adequate mechanical properties, along with high biocompatibility and the ability to integrate with bone tissue. This study compares the corrosion behavior of heat treated Ti-12Mo-13Nb alloy at 950°C/1h, Ti-12Mo-13Nb as cast, commercial pure Ti (cp-Ti) and Ti-6Al-4V alloys by potentiodynamic polarization tests. The Ti-12Mo-13Nb alloy ingots were produced using an arc-melting furnace with non-consumable tungsten electrode under argon atmosphere. Corrosion resistance tests were carried out in Ringer's solution (pH = 7.0) using an electrochemical cell set up with a working electrode (samples), a counter electrode (Pt) and a standard calomel as the reference electrode. The electrochemical tests were conducted using potentiodynamic polarization applying potentials from -2000 mV to +2000 mV in relation to the open circuit potential (OCP). From the polarization curves, the corrosion potential, pitting potential and corrosion current were determined. The results indicated that the Ti-12Mo-13Nb alloys is less susceptible to corrosion mechanisms compared to commercially pure Ti (control) alloys. Therefore, the Ti-12Mo-13Nb alloys can be considered as an interesting alternative for biomedical applications, as it does not contain potentially cytotoxic elements and showed satisfactory anti-corrosive properties in electrochemical test.

[1] Chui, P.; Jing, R., Zhang, F.; Li, J.; Feng, T. Mechanical properties and corrosion behavior of β-type Ti-Zr-Nb-Mo alloys for biomedical application. J. Alloys Compd, 842 (2020).

Computational study of a binuclear coordinationcomplex of copper(II) with maleate and 1,10- phenanthroline ligands

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Coordination complexes of transition metals containing mixed ligands have been studied intensively due to their promising applications in different fields [1]. Therefore, the development of computational studies of these complexes is also important and has been performed using density functional theory (DFT) [2]. The objective of this work was to develop a theoretical study of the geometrical, thermodynamical, and electronic properties of the complex of Cu(II) with maleate and 1,10-phenanthroline ligands. The software Gaussian 16 was used for the development of the electronic structure calculations, employing the DFT functional PBE1PBE combined with the 6-311++G(d,p) for H, N, O, and C atoms, using the SDD pseudopotential and basis set for the Cu atoms. From the optimized geometry obtained in a triplet state, a slightly distorted square pyramidal geometry is observed around each metal center, in good agreement with experimental results [3]. Calculated values of Gibbs free energy and enthalpy of the complexation are negative, indicating that the process is spontaneous and exothermic. The HOMO and LUMO are concentrated over the maleate and phenanthroline ligands, respectively. The gap between the β-HOMO (the true HOMO) and LUMO is 4.69 eV and this value was used for chemical reactivity indices such as chemical potential, hardness, softness and electrophilicity, that indicated that the electronic structure of the complex is stable. The computational results showed the binuclear coordination structure of Cu(II) and indicated that the complexation reaction is spontaneous and exothermic.

Acknowledgments: We thank the FAPEMA, CAPES, CNPQ for supporting this research.

References:

[1] Y. Q. Zheng, X. S. Zhai, L. J., H. L. Zhu, et. al, Polyhed. v. 68, p. 324, 2014.

[2] H. Chermette, Coord. Chem. Rev. v. 178, p. 699, 1998.

[3] Z. Y. Li, D. J. Xu, J. Y. Wu, M. Y. Chiang, Jour. Chem. Cryst., v. 35, p. 615, 2005.

Converting Waste Materials into Biomaterials: Enriched calcium zeolites synthed from chickeneggshells for hemostatic applications

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Abstract

Although there is a plethora of 245 different zeolites with different frameworks with unique and unusual topologies of channels and cavities with large applications in the field of industrial catalysis [1], few of them have found systematic biomedical applications, especially for the purpose of hemorrhage control. Hemostasis is a complex biochemical process in which the assembly of substrates, enzymes, protein cofactors and calcium ions ona phospholipid surface accelerates the rate of coagulation. Several inorganic solid matrices have been employed as topic hemostatic agents, and among them zeolites [2]. However, the synthesis of enriched calcium zeolites for the purpose of hemorrhage control and using as raw materials chicken eggshell biomass is an unexplored field of research. We report the data of the synthesis of four calcium zeolites using $CaCO3$ derivated from chicken eggshell. Thromboelastographic results using the pristine calcium enriched zeolites show that they were able to promote a faster clot formation and a higher maximum clot strength than those of the commercial topical hemostatic agent QuikClot.

Acknowledgements

FAPESP, CAPES and CNPq have supported this work.

References

[1] Fernandez, S., Ostraat, M. L., Zhang, K. (2020). Toward rational design of hierarchical beta zeolites: An overview and beyond. In AIChE Journal (Vol. 66, Issue 9). Wiley. https://doi.org/10.1002/aic.16943

[2] Malik, A., Rehman, F. U., Shah, K. U., Naz, S. S., Qaisar, S. (2021). Hemostatic strategies for uncontrolled bleeding: A comprehensive update. In Journal of Biomedical Materials Research Part B: Applied Biomaterials (Vol. 109, Issue 10, pp. 1465-1477). Wiley. https://doi.org/10.1002/jbm.b.34806

Corrosion resistance and structural characterization ofa titanium β alloy processed by angle channel angular pressing

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Titanium and its alloys are widely used for implants manufacturing and biomedical devices, due to their high corrosion resistance and biocompatibility. In this work, the study of the corrosion resistance and structural characterization of a Ti-29Nb-13Ta-4Mo β-Ti alloy was carried out. The alloy was investigated in as-received state and after several passes of equal channel angular pressing (ECAP). In ECAP, a metallic specimen is pressed though a metallic die containing two channels, equal in cross section area, intersecting at a die angle, where it is constrained and bent through a sharp angle which imposes high plastic shear strain in material without any reduction in cross-section area. ECAP was performed using a die with 120º between the channels. The processing was performed at 350 ºC up to 8 ECAP passesby route A, into which the specimen is not rotated between each ECAP pass. The sampleshad their microstructures analyzed by SEM/EBSD and XRD while the corrosion resistancewas evaluated by potentiodynamic polarization tests. The interplay between the ECAP processing, microstructure and corrosion performance is discussed in this work.

Acknowledgements:
Authors acknowledge the financial support from the FAPESP (Grants 19/22183-6) and CNPq (Grant: 310984/2020-3). The Multiuser Central Facilities of the Federal University of ABC (CEM-UFABC) is also acknowledged for facilities access.

Cryobioprinting of Gelma Scaffolds Mechanically Reinforced by Multi-layer PVA Electrospun Nanofibers

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Among the manufacturing techniques to regenerate damaged tissues are electrospinning, widely used to make nanofibrous muscle mimetic supports for tissue regeneration, and 3D bioprinting has also been used to manufacture muscle tissues in recent times [1]. Cartilage has a very limited ability to self-repair after traumatic injuries, representing a heavy socioeconomic burden. Regenerative biomaterials can create a favorable local environment for cartilage repair without causing relevant regulatory and scientific concerns of cell-based treatments [2]. Gelatin methacrylate (GelMA) is a versatile matrix that can be used to create tissue structures ranging from vasculature to cartilage and bone, has good biocompatibility, solubility and degradability [3]. PVA is a cheap, water-soluble, non-toxic and biocompatible polymer, often used in biomedical applications. For these reasons, and due to its excellent wiring capacity, PVA is often considered to be excellent in microfribas electrowiring processes [4]. Based on the above, this work aimed to develop a biomaterial structured in alternating layers between electronically rotated PVA fibers and bioprinted GelMA at low temperatures, creating a multilayer scaffold. Characterizing the material with mechanical, chemical and biological tests, thus achieving a scaffold with good mechanical strength higher than 15kPa compatible with the inserted medium and optimal cell viability.

Acknowledgements: CAPES, CNPq and LIMAV

References: Thangadurai, M; et al. Advances in electrospinning and 3D bioprinting strategies to enhance functional regeneration of skeletal muscle tissue. Biomaterials Advances, p. 213135, 2022.

2. Zhou L, et al Engineered biochemical cues of regenerative biomaterials to enhance endogenous stem/progenitor cells (ESPCs)-mediated articular cartilage repair. Bioactive Materials, 2023.

3. Piao, Y., et al. Biomedical applications of gelatin methacryloyl hydrogels. Engineered Regeneration, 2021.

Development of a borohydride-free methodology to prepare silver nanoparticles

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Nanoparticles have been used in several applications, due to unique properties, such as elevated surface area, conductivity, small size, and optical features [1]. Namely, silver nanoparticles (AgNPs) have attracted much interest in biomedical applications due to their anti-inflammatory and antibacterial properties, which provide excellent healing inactivating various microorganisms. Additionally, AgNPs are widely used to produce biomaterials that will be in direct contact with the human body, however, for their synthesis, the main reducing agent used is sodium borohydride($NaBH_4$), wich presents high toxicity[2]. Therefore, this project aims to develop a more biocompatible synthetic route to prepare biomedical applications. For this, sodium citrate was used as a stabilizer, $AgNO_3$ as a silver source, and ascorbic acid as a reducing agent. The pH was maintained at 11 and the reaction occurred at 50 ºC for 30 minutes. The samples were characterized by UV-Vis spectroscopy, and the spectra showed only one band 410, indicating the formation of spherical AgNPs. However, the absorbance presents a lower intensity, varying form 0.6 to 1.0, when compared to the AgNPs formed in the presence of $NaBH_4$ (absorbance around 2.0), indicating a lower concentration of AgNPs. Thus, more tests will be made to optimize procedure, testing different stabilizers and physicochemical parameters. Nevertheless, our results indicate that this synthetic route is promising to obtain more biocompatible AgNPs, ideal for biomedical applications.

Acknowledgements:

The authors thank the UFPE, PIBIC/CNPq (220118038), FACEPE (IBPG-1324-1.06/21) and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020).

References

[1] Miyazaki and Islam, Technovation, 2007, 661, 101016.

[2] Monte, J.; et al., Materials Letters, 2023, 338, 134027.

Development of a scaffold based on the synthesis of monoacylglyceride from buruti oil (*Mauritia flexuosa*)for bone tissue engineering.

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Bone diseases and fractures are among the most common orthopedic problems and represent an important global health problem and have been the subject of current studies. The standard clinical treatment used to repair fractures and bone defects is bone grafting (autograft, allograft), but they face major challenges due to the lack of donors for bone transplantation, costs, and risk of disease transmission [1]. Thus, it is essential to develop an alternative device, capable of mimicking the characteristics of the extracellular matrix, providing an appropriate microenvironment to facilitate the organization of cells, and thus, control and direct the formation of a new tissue, which is the objective of this work [2]. A porous bioactive scaffold was synthesized from monoacylglyceride (MAG), derived from buriti oil (Mauritia flexuosa). Buriti oil (BO) contains a high concentration of monounsaturated fatty acids, carotenoids, polyphenols, and bioactive substances with high antioxidant and healing capacity, which makes it an excellent raw material for tissue engineering. The scaffold synthesized from the reaction between MAG, obtained by glycerolysis reaction, and polyethylene glycol (PEG) in the presence of hexamethylene diisocyanate (HDI) was confirmed by FT-IR, thermal stability by TG and DSC, morphological evaluation by SEM and the presence of BO components confirmed by UV-VIS. Acknowledgment: SEDUC-PI, UFPI, LIMAV. [1] Lima, F. S.; Matos, L. F. L.; Pacheco, I. K. C.; Reis, F. S.; Pierot, J.; Camara, J. V.; Matos, J.M.E.; Ribeiro, A. B.; Moura, W. L.; Fialho, A. C. Scaffold based on castor oil as an osteoconductive matrix in bone repair: biocompatibility analysis. Polímeros: ciência e Tecnologia, v. 32, p. e2022003 1-e2022003 5, (2022). [2] Reis LVC, Leão KMM, Ribeiro APB, de Jesus MB, Macedo GA, Macedo JA. Evaluation of cytotoxicity of nanolipid carriers with structured Buriti oil in the Caco-2 and HepG2 cell lines. Bioprocess Biosyst Eng. p.1105-1118, (2020).

Development of zinc alloys by powder metallurgy for application as biomaterial

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Biodegradable metals have been developed for application as biomaterials with the advantages of gradual degradation, avoiding secondary surgeries and offering better mechanical properties when compared to ceramic and polymer materials [1]. Zinc is a daily essential chemical for the human body and plays a key role in human health. The human body requires about 15 mg/day of zinc, which proves that zinc has good biocompatibility for the human body and is therefore safe for biomedical applications, in addition to being considered a biodegradable metal [2]. Synthesis by powder metallurgy and more, the study of mechanical properties, microstructure and composition of zinc matrix alloys was carried out by Differential Scanning Calorimetry (DSC), to define alloy sintering temperature, Vickers hardness, X-ray diffraction (DRX), X-ray fluorescence and microtomography respectively. The DSC analysis indicated the sintering temperature of the samples that were synthed with a heating rate of 15 K/min to 553 K for 2 hours and a natural cooling stage of 553 K to room temperature. XRDs reveal a zinc matrix with a compact hexagonal crystalline structure of space group P63/mmc. Furthermore, the hardness tests show an increase in hardness with an increase in the concentration of the dopant, boron and a decrease with an increase in the porosity profile of the samples, a profile obtained by microtomography.

Acknowledgments
The Federal District Research Support Foundation (FAPDF), the University of Brasilia (UnB) and the Federal Institute of Pará (IFPA) for encouraging research.

References

[1] Hu, Y., Guo, X., Qiao, Y., Wang, X., Lin, Q. Preparation of medical Mg-Zn alloys and the effect of different zinc contents on the alloy. Journal of Materials Science: Materials in Medicine, v. 33, n. 1, p. 1-13, 2022.

[2] Hernández-Escobar, D., Champagne, S., Yilmazer, H., Dikici, B., Boehlert, C. J., Hermawan, H. Current status and perspectives of zinc-based absorbable all

Direct observation of the transformation of amorphous calcium carbonate to calcite using cryo-transmission electron microscopy techniques

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Calcium carbonate ($CaCO_3$) is a frequently used model mineral in studies of crystallization mechanisms. Where the diversity of the origins, composition, morphologies, and polymorphs of $CaCO_3$ makes it a material with high potential in applications biotechnological. Crystalline $CaCO_3$ exhibits three polymorphs: hexagonal vaterite, orthorhombic aragonite, and rhombohedral calcite, in order of increasing thermodynamic stability. The physical properties of $CaCO_3$, such as structure, shape and polymorphism, is strongly correlated with the crystallization pathways underlying their formation [1]. Due to the stochastic nature of nucleation and growth, the effective route to understanding is to directly capture the building blocks as they interact and crystallize. In this work, a systematic study of the different steps of $CaCO_3$synthesis was performed using cryo-transmission electron microscopy (Cryo-TEM) in the modes of HRTEM, SAED, EDS and EELS. This technique allows preservation of 3 µL of the specimen by vitrification at different stages of the synthesis process, preventing their modification during drying. For the synthesis of $CaCO_3$we use $CaCl_2.2H_2O$ (1M) and Na_2CO_3(1M) what were obtained from Sigma Aldrich. Aqueous solutions of the reactants was made using milli-Q water. In vitro evidence is provided for the transformation of amorphous calcium carbonate (ACC) to calcite throughthe mechanism of dissolution-reprecipitation. Therefore, using a synthetic system, we provide evidence for the transformation of ACC into calcite in highly supersaturated solution. While our reaction conditions are significantly different from those used by organisms during biomineralization, our results demonstrate that this non-classical pathwayis a possible route for the precipitation of the thermodynamically more stable phase of $CaCO_3$.

ACKNOWLEDGEMENTS: The authors would like to thank CNPQ, FAPERJ and LABNANO/CBPF.

[1] Zihao Ou, at. al., nature materials. v19, pages 450-455 (2020).

Effects of bisphenol-A (BPA) on giant unilamellar vesicles (GUVs) composed by DOPC/cholesterol appliedas mimetic system of cell membrane

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Bisphenol-A (BPA), present in the composition of polycarbonate plastics and epoxy resins, the main materials of household items such as cups and bottles, is considered an emerging pollutant since it affects the human endocrine system, influencing the action of hormones in the body [1]. However, the health risks of chronic exposure to BPA are not yet understood in detail. An efficient way to obtain conclusions about the effects of compounds such as BPA on the body is the study of its interaction with mimetic systems of cell membranes (composition and), such as lipid vesicles. Here we investigated the action of BPA on giant unilamellar vesicles (GUVs) formed by a mixture of the phospholipid DOPC and cholesterol, ensuring both chemical composition and fluidity of eukaryotic cells. The GUVs composed by DOPC/cholesterol in the ratio 8/2 in mol were formed by electroformation and analyzed under a confocal microscope. When exposed to BPA, the GUVs presented fluctuations and several morphological deformations, such as: i) change of the spherical shape, ii) bud transition (formation of two smaller vesicles from a larger vesicle), and iii) formation of lipid cords. The morphological changes were observed within 2 minutes after the addition of BPA to the GUVs, with the contaminant causing a stress on the curvature of the vesicles, disturbing the integrity of the membrane. Therefore, this work highlights the importance of understanding the effect of BPA on structures that preserve both the and the curvature of the cell membranes, being suitable indicators of its action on biological processes.

Acknowledgments:

FAPESP 2018/22214-6, CNPq, CAPES, INCT/INEO.

Reference:

[1] L. Trasande. Further Limiting Bisphenol A In Food Uses Could Provide Health And Economic Benefits. Health Aff 33 (2014) 316-323. DOI: 10.1377/hlthaff.2013.0686.

Effects of CoCr-enriched medium on shear-stressed endothelial cell and osteoblasts

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Cobalt-chromium (CoCr) based alloys are biocompatible, resistant to corrosion and non-magnetic, turning them interesting as biomaterials in the biomedical field [1]. However, little is known about its effect on cell metabolism, which motivated this study. Firstly, CoCr discs [subjecting (W) or not (Wo) to dual acid-etched (DAE)] were incubated into FBS - free cell culture medium up to 24 h (37 °C). This CoCr-enriched medium was further used to treat shear-stressed endothelial cell cultures up to 72 h. Thereafter, the conditioned medium containing metabolites of shear-stressed endothelial cells in response to CoCr-enriched medium was further used to subject osteoblast's cultures, when the samples were properly harvested to allow the analysis of the molecular issues. The CoCr-enriched medium was found to modulate the metabolic pathways of both cells, inducing the expression of genes such as HIF1α, VEGF, nNOS, osteocalcin and bone sialoprotein. Furthermore, shear stresson endothelial cells increases the activity of matrix metalloproteinases in bone cells [1,2]. Altogether, our data shows for the first time that shear-stressed endothelial metabolites responding to CoCr discs contribute to osteogenic phenotype *in vitro*, and this predicts an active crosstalk between angiogenesis and osteogenesis during osseointegration of CoCr alloy and bone healing, maybe guided by the Co-induced hypoxic condition.

Acknowledgements: CAPES, FAPESP and CNPq.

References:

1. Machado, M.I.P. et al. Cobalt-Chromium-Enriched Medium Ameliorates Shear-Stressed Endothelial Cell Performance. J. Trace Elem. Med. Biol, 54, 163-171. 2019
2. da Costa Fernandes et al. Metabolic Effects of CoCr-Enriched Medium on Shear-Stressed Endothelial Cell and Osteoblasts: A Possible Mechanism Involving a Hypoxic Condition on Bone Healing. Mater. Sci. Eng. C, 128, 2021.

ELECTRODEPOSITION OF APATITES ONTi-35Nb-7Zr-5Ta ALLOYS

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In recent decades, biomaterials have become increasingly important in the field of medicine. Being used to supply lost or diseased biological structures to improve the quality of life of the patient [1]. HAp electrodeposition on titanium alloys has shown to be a promising technique to improve the biocompatibility and osseointegration of orthopedic implants [2]. The present work aims to analyze the deposition of apatite on the Ti-35Nb-7Zr-5Ta alloy via electrodeposition. The HAp solution was prepared, and the process was carried out for two different periods: for 2h and 4h with heating at 70 °C. In both Ramann Spectroscopy analyzes, both for 2h and 4h, the peak at 960 cm-1 is noted, being a typical point for calcium phosphate, attributed to the vibrations of the phosphate and hydroxyl present in the hydroxyapatite. EDS analysis of HA-Ti-35Nb-7Zr-5Ta confirmed the presence of titanium, calcium, phosphorus, tantalum, sodium and niobium for both the 2h and 4h processes. This composite biomaterial is known for its excellent biocompatibility and mechanical properties. Titanium is widely used in medical applications because of its biocompatibility and corrosion resistance. Calcium and phosphorus are essential components of hydroxyapatite, which is a natural material found in human bones. Tantalum and niobium are refractory metals with high resistance to corrosion and oxidation. It was shown that the results obtained by SEM show that, during the periods of 2h, 4h there was the formation of tetracalcium phosphate (TTCP), it can be noticed the elongation of the apatite with the increase of the period. In addition, it was possible to observe, by the morphological aspect of the surface, the gradual and constant transformation of the material. And, with the effectiveness of the deposition of apatite in the alloy, we can see the excellent biocompatibility of the material.

[1] HE, C. Hydroxyapatite Nanoparticles for Biomedical Applications, 2019.

ELECTROSPINNING OF Ag NANOPARTICLES/PVA NANOFIBERS FOR BIOMEDICAL APPLICATIONS

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The interest in studies involving the development of intelligent wound dressings for chronic wounds has been gradually growing, since they constitute a serious public health problem. In this context, nanofibers are highly attractive due to their characteristics, such as low cost, high surface-to-volume ratio, porosity, flexibility and simple processing [1]. Furthermore, the possibility of combination with different compositions and properties allows for varied applications. In particular, poly (vinyl alcohol) (PVA), a semi-crystalline, hydrophilic polymer, widely used due to its biocompatibility and biodegradability properties [2] In the present work, silver nanoparticles were synthesized by the chemical reduction method by salts and incorporated into PVA fibers, which were obtained by electrospinning. The physicochemical and morphological properties of the nanofibers were investigated.Scanning electron microscopy (SEM) images indicated nanofibers with homogeneity and nanometric diameter. Furthermore, it was possible to observe an average diameter of 68.57 nm for silver nanoparticles (Ag-NP) that, when incorporated into the electrospun PVA mats, remained at nanometric scales, which can be evaluated by SEM and Energy Dispersion X-Ray Spectroscopy (EDS). It was also possible to observe that Ag-NP incorporated at lower concentrations into PVA nanofibers demonstrated rapid biodegradability, indicating their potential for application where immediate releases are required. Therefore, Ag-NP/PVA nanofibers can be an interesting alternative for use in biomedical applications.

Acknowledgements:CAPES

References:

[1] Xue, J. et al. Electrospinning and Electrospun Nanofibers: Methods, Materials, and Applications. Chemical Rev., v. 119, n. 8, p. 5298-5415, 2019.

[2] Jin, S. G. Production and Application of Biomaterials Based on Polyvinyl alcohol (PVA) as Wound Dressing. Chem Asian J., v. 17, n. 21, 2022.

Electrospinning of type I collagen and PVA nanofibers using a green solvent

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Electrospun collagen nanofibers can mimic the architecture of native human tissues, making them an attractive option for tissue engineering applications [1]. Collagen nanofibers are inherently mechanically weak, which may limit their use in load-bearing applications [2]. To overcome this limitation, collagen is commonly cross-linked or blended with synthetic polymers to improve mechanical strength without compromising biological activity [3]. In this work, we report the use of the ecological solvent acetic acid for electrospinning of the type I collagen blend from bovine skin with polyvinyl alcohol (PVA) with the aim of increasing the mechanical strength of the resulting scaffold and preserving the biological activity of the collagen. Characterizations of the polymers and membranes were performed by Fourier Transforms Infrared (FTIR), X-ray Diffraction Analysis (XRD) and Scanning electron microscopy (SEM). The results indicated that the presence of collagen results in samples with a less crystalline structure. Blending PVA with collagen improved the spinning property and mechanical properties of the composite fibers. The PVA-Collagen membrane exhibits an FTIR spectrum that indicates the PVA and collagen components by comparing their respective wave numbers. The characteristics of the collagen bands, Amide I, II and III and the acetate group of PVA were identified. Nanofibers in the range of 100-200 nm, whereas solid-state characterization showed the successful integration of collagen with PVA.

Acknowledgements:

The authors are grateful for the financial support of the State of São Paulo Research Foundation (FAPESP), CAPES and the Aeronautics Technological Institute.

Reference

[1] S. Anjum et al., Int. J. Mol. Sci., vol. 23, no 16, p. 9206, ago. 2022.
[2] J. X. Law et al., Tissue Eng. Regen. Med., vol. 14, no 6, p. 699-718, ago. 2017.
[3] Y.-H. Jiang et al., Am. J. Transl. Res., vol. 14, no 2, p. 1146-1159, fev. 2022.

Emissive complex between 4'-N,N-diethylaminoflavonolin octa-acid deep-cavity cavitand and Rhodamine 6G

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The investigation of the chemistry of the excited state plays a crucial role in the field, as it relies on intermolecular communication. This aspect holds significant importance in materials science research. To explore the interaction in such systems, we investigated the ground and excited states of 4'-N,N-diethylaminoflavonol (DEA3HF) in an octa acid-based (OA) confined medium in the presence of Rhodamine 6G (R6G). It is worth mentioning that the solubility of organic compounds is often limited to organic solutions, so the use of the supramolecular approach, which involves confining them in a capsule without affecting their photophysical properties, significantly expands the study and applications [1]. For this purpose, fluorimetric titrations were performed using steady-state and time-resolved fluorescence spectroscopy in different environments. Despite the observed spectral overlap between the flavonol emission and the R6G absorption, as well as the fluorescence quenching of the flavonol in the presence of R6G, the almost constant fluorescence lifetimeat different amounts of R6G discards the presence of FRET. Steady-state and time-resolved fluorescence indicate the formation of an emissive complex between the proton transfer dye encapsulated within water-soluble supramolecular host octa acid (DEA3HF@(OA)2) and R6G. The respective Stern-Volmer plots corroborate these observations, suggesting a static quenching mechanism, which the formation of an emissive complex in the ground state.[2]

Acknowledgments: CNPq, PPGQ-UFRGS

References:

[1] Gupta, S.; Adhikari, A.; Mandal, A.K.; Bhattacharyya, K.; Ramamurthy, V. J. Phys. Chem. C 115., 9593-9600 (2011)

[2] Santos, F.S.; Ramasamy, E.; da Luz, L.C.; Ramamurthy, V.; Rodembusch, F.S. Molecules 28, 4260 (2023)

Encapsulation of curcumin in nanogels for thetreatment of cutaneous leishmaniasis

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Nanogels are promising drug delivery systems for the treatment of different diseases, being of great interest for new alternative antiparasitic therapies, in order to overcome the various limitations found in current chemotherapies for leishmaniasis, such as high toxicity and resistance. Curcumin (CUR) has a vast antiparasitic activity, however, its applications are limited, since it is an unstable molecule, which can be degraded by light and temperature, in addition, the low solubility in water reduces its bioavailability [1]. Despite some biological limitations regarding its absorption in the body, encapsulation of curcumin in nanogels based on epoxy polyetheramine, are non-toxic, biodegradable, biocompatible and represent an effective strategy to deliver CUR in a controlled and selective manner. The aim of the present study was to develop an effective, non-toxic and biodegradable polymeric drug delivery system encapsulating curcumin for the treatment of cutaneous leishmaniasis. the specificity of nanogels. Nanogels showed advantages in CUR transport and release, including increasing the stability of the molecule and delaying its degradation.

Reference: [1] Tian Jiang, Wei Liao, Catherine Charcosset. Recent advances in encapsulation of curcumin in nanoemulsions: A review of encapsulation technologies, bioaccessibility and applications. 132, 2020. Food Research International.

Acknowledgment: The authors are grateful for financial support provided by FAPESP (grants #2021/06552-1, #2022/06507-9, #2022/13408-7 and EMU #2021/14619-9), CAPES (Finance Code 001), and CNPq (grant #307696/2021-9).

Engineering of Polymer Vesicles with Selective Permeability towards Potential Applications in the Field of Biotechnology

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Polymersomes are a type of synthetic vesicles formed by a polymer membrane that surrounds an aqueous inner cavity. Their colloidal stability and membrane permeability determine their potential applications,[1] including drug delivery and biochemical reactions in confined spaces.[2] The membrane permeability is typically governed by the chemical nature and length of the hydrophobic segment. In this work, we investigated the self-assembly of a variety of diblock copolymers using thin film hydration (FH) and solvent-switch (SS) approaches. Accordingly, the block copolymers were dissolved in a suitable organic solvent. In the case of FH, the solvent was evaporated, and the formed thin polymer film was hydrated using PBS solution. As for SS, PBS was dropwise added into the organic polymer solution. The systems obtained were characterized by dynamic (DLS), static (SLS) and electrophoretic light scattering. In all cases 0.2 mg/ml polymerosome solutions in PBS were measured. SLS was measured from 30 to 150° with 5° stepwise increase and DLS was measured at 90°. Nanoparticles with ranging from 75 to 160 nm were obtained, and the structure sensitive parameter ρ= RG/RH~1 suggested the formation of polymer vesicles. This is also corroborated by the determined values of Nagg (Nagg~10^3) commonly obtained for polymersomes. We have ongoing research to study the permeability of different polymersomes to different analytes. For small molecules such as rhodamine, we conduct encapsulation and release assays. We also investigate their permeability to solvents via light-scattering experiments, and we are simultaneously exploring the proton permeability using pH-responsive probes.

Acknowledgements: We acknowledge the sponsoring by FAPESP (grants 2019/06634-8 and 2021/12071-6). A. P. R. acknowledges the fellowship granted by FAPESP (grant 2022/14668-2).

References:

[1] A. J. Miller et al., ACS Cent. Sci., 7, 30-38 (2021)
[2] L. D. Blackman et al., ACS Macro Lett., 6, 1263-1267 (2017)

Evaluation of osteogenic and antimicrobial propertiesof niobium nanocomposites

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Nanotechnology presents a breakthrough in the discovery and exploration of the physical and chemical properties of new promising materials for numerous scientific applications. The use of nanomaterials applied to bone regeneration, for example, have become the target of studies in bone tissue engineering. In particular, nanomaterials based on niobium such as polyoxoniobates (PNb) have been attracting great interest due to their high solubility in water, low toxicity on the human body and provide greater bioactivity of bone cells. The PNb´s have osteoinductive properties, being indicated as a material that allows to promote osteogenesis and angiogenesis, and, therefore, promising to repair defects and favor osseointegration.

Recent research shows that polyoxoniobates have versatile properties due to their antimicrobial action against resistant organisms, bioactivity, high levels of adhesion and osteoblasts proliferation [2]. Thus, the objective of this work was to associate the antimicrobial and osteogenic properties of the Nb nanocomposite to favor and improve osseointegration. The physicochemical properties of the material were investigated using SEM, FTIR, TG, RAMAN, DLS, and in vitro assays to assess osteogenic, antimicrobial, and antioxidant activity.

Acknowledgements: CDTN,CNPq, FAPEMIG, Finep.

References:
[1] PEREIRA-MAIA, E. C. et al., RSC Advances 19 (2018) 10310.
[2] ARAUJO, R. C. Fosfatos de cálcio modificados com nióbio e seus compósitos com niobatos lamelares aplicados como agentes antimicrobianos e adsorventes, Tese (Doutorado), Química, UFPB, 2022.

Evaluation of PVA hydrogel loaded with Apitoxin for treating skin cancer—preliminary data

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Recently, interest has grown in treating cancer with natural products, including bee venom or aptoxin (APX). APX, due to its main constituent, melittin, induces cytotoxic, antitumor, immunomodulatory and apoptotic effects in different cells in vitro and in vivo. However, the toxicity of apitoxin and the nonspecific hemolytic effects make its clinical application unfeasible. Hydrogels are materials capable of absorbing and releasing large amounts of fluids according to predetermined factors such as pH, temperature, time, among others. Polyvinyl alcohol hydrogel (HPVA) is a new material that can be an interesting alternative for drug administration due to its biocompatibility and ability to absorb and release substances with both positive and negative charges. In addition, it is a chemically inert material, which are the main characteristics for the development of a platform (dressing) for drug release. Melanoma is the most serious skin cancer with high mortality rates due to its highly invasive and aggressive nature. The search for new topical treatments can collaborate with the local treatment of this type of cancer with less systemic effects. Thus, this work evaluated the potential for absorption and release of APX conjugated to HPVA for future analyzes using cultures in two and three dimensions (2D and 3D). HPVA was characterized by FTIR and SEM. The apitoxin was diluted in PBS at a concentration of 2.5 mg/mL and incubated with 10 mg of HPVA at 37 ºC for 48 h. The ability to absorb and release the main components of APX (melittin and phospholipase) was evaluated by UV-Vis at different times. The best results were observed after 48 hours of incubation, where 44% of the melittin and 50.7% of the phospholipase present in the solution were absorbed by the HPVA. Release was on average 50% of both melittin and phospholipase absorbed. The results are preliminary, however the HPVA/APX presents excellent possibilities to composea curative for the treatment of melanoma.

Exploring the photothermal efficiency of gold-shell isolated nanoparticles on triple negative breast cancer cells

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With 2.3 million new cases per year, breast cancer stands as the top malignancy affecting women worldwide. In this scenario, the growing occurrence of triple negative breast tumors poses a significant challenge for treating patients. Triple negative cancer cells do not express the receptors estrogen (ER), progesterone (PR), and human epidermal growth factor 2 (HER2), being therefore, more aggressive and difficult to treat than other cancer subtypes. In the search for more efficient, less invasive and low side effect treatments, photothermal therapy (PTT) has drawn attention. Photothermal agents convert light into heat, leading to cell death via hyperthermia and can generate reactive oxygen species(ROS), which induce oxidative stress in the cell membranes. Herein, gold shell-isolated nanoparticles (AuSHINs) are applied as photothermal agents to target MDA-MB-231 cells, a human breast adenocarcinoma cell line derived from metastatic breast cancer patients and classified as triple-negative cancer cells. AuSHINs incorporation and cellular localization was assessed by flow cytometry and confocal fluorescence microscopy, respectively. The photo-efficiency of AuSHINs is more reliant on light dose than incubation time. When exposed to 20 and 30 min of irradiation, the viability of cells incubated with AuSHINs for 0.5h decreases from ca. 77% to 16%, while for 2.0 h of incubation, the viability decreases fromca. 70% to 12%. The light doses also affected the cell death pathways. After 20 min irradiation, most of the non-viable cells are in early apoptosis. In contrast, after 30 min, the cells progress to late apoptosis, and the viable cells decrease drastically. Intracellular ROS was also detected and found to increase with the incubation time. These findings can help in comprehending the response of triple-negative breast cancer cells to PTT and may lead to the exploration of novel treatment strategies.Acknowledgements: CNPq, FAPESP, and the Crafoord Foundation.

Facile glycerol-isopropanol synthesis route of nanostructured $Mg(OH)_2$ and MgO doped with Calcium-based nanoparticles

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New research advances in bone tissue regeneration showed a key role in ion-doped biomaterials' function with ceramic scaffolds. The addition of Ca ions is essential to supply Ca^{2+} ions to/from the cellular media, also magnesium Mg^{2+} ions stimulate osseointegration by increasing bone-implant contact, push-out force, and new bone formation [1]. In this work, we report the synthesis and characterization of $Mg(OH)_2$ and MgO nanoparticles (NPs) doped with calcium for biological applications. The glycerol-urea (GU) method was developed by our research group, and it is an innovative and efficient method for the synthesis of metallic hydroxy/oxides [3], although for this study the urea was replaced by isopropanol (GI) for better conditions for later large-scale production. The syntheses were carried out using $Mg(NO_3)_2{\cdot}6H_2O$ and NaOH in a 3:1 mol ratio of G:I solution under stirring at 70°C, with addition of 0.001, 0.005, 0.01 and 0.05 mols of $CaCl_2{\cdot}2H_2O$. To obtain MgO,the samples were calcined in a muffle oven for 3 hours in 400°C. Structural characterizationwas performed by X-Ray Diffraction. Results showed formations of brucite ($Mg(OH)_2$) and calcium carbonate ($CaCO_3$), with crystallites of 10 and 100 nm respectively, this formation has several advantages for biomedical applications, such as: biocompatibility, sensitivity to pH, low cost, biodegradability, and low cytotoxicity [3]. TEM and SEM images showed NPs' morphology of sheets and shells with high surface area. ICP-OES data will be used to assess the amount of calcium ion release from NPs. These initial results already show the great potential of this material for biomedical applications.

Acknowledgements: Unifesp, EEL-USP and Capes and Fapesp Grant n° 2021/01509-0

References:

[1] S.S. Lim, et al, Mater. Sci. Eng. C Mater. Biol. Appl., vol.76, 144-152 (2017).
[2] Brazuna, et al. New J. Chem., vol. 43, 18988-18995 (2019).
[3] MEMAR, et al. Current Microbiology, vol. 80, 1-11 (2023).

Fatigue behavior effect of shot peening associated to plasma nitriding surface treatment in F138 stainlesssteel

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Analysis of shot peening associated to plasma nitriding surface layers on austenitic stainless steel are conduced to verify the influence of the surface treatment in fatigue behavior. Shot peening can be used to increase fatigue resistance, due to the surface residual stress induced. Additionally, shot peening can be used as a previous plasma nitriding treatment to improve nitrogen atoms diffusion, increasing the layer hardness, thickness and fatigue resistance [1]. Thus, the aim of the present work is to study the effect of previous shot peening surface treatment in plasma nitrided F138 stainless steel fatigue properties. Specimens treated with plasma nitriding and shot peening associated to plasma nitriding were analyzed in high cycle fatigue tests. Previous shot peening treatment to plasma nitriding promoted a failure in lower strength values than reference specimens, and its similar to plasma nitride specimens. The result can be associated to roughness increase due to shot peening and plasma nitriding treatments, which can influence fatigue behavior as other surficial morphological characteristics of the formed layer [2]. Fracture surface were analyzed by scanning electron microscopy (SEM) and ductile fracture surface was observed in all conditions. In higher load conditions cracks and microcraks were formed on the sides of the specimen.

Acknowledgements:
CAPES, IPEN, IPT, USP, Sandvik Coromant, SintoKogio, TTI Plasma e Villares Metals.

References:

[1] SHEN, L., WANG, L., WANG, Y., WANG, C., 2010, “Plasma nitriding of AISI 304 austenitic stainless steel with pre-shot peening”, Surf. Coat. Technol., Vol. 204, pp. 3222-3227.

[2] WIESER, H., 2004, “Influence of shot peening on the corrosion fatigue properties of a stainless steel for surgical implants”, Materials and Corrosion, Vol. 55, pp. 186-193.

Formation and antibacterial action of hybrids based on Nerolidol and bentonite

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Essential oils are molecules with intense bioactivity, and their uses in several fields of industry, such as health and food, have been explored [1]. However, the high volatility of essential oils limits their use [1,2]. Therefore, incorporating essential oils in solid materials is a strategy to maintain their properties. This study investigated the incorporation of Nerolidol at 50% v/v concentration in raw and organophilic bentonites. The antibacterial action of the oil/clay hybrids against *Staphylococcus aureus*and *Escherichia coli*was also evaluated using the direct contact test. XRD patterns confirmed the intercalation of the oil in sodium bentonite, increasing the basal spacing of 1.26-1.50 nm, and in the cetyltrimethylammonium clay (CTAB-Bent), which value was 3 nm. FTIR spectra concord with the interaction between the oil and OH and organic groups in raw and organophilic samples, respectively. The antibacterial of the hybrids revealed 100% inhibition for both bacteria. Promising results indicate that obtained hybrids materials can be used in cosmetics and health products.

Acknowledgments: CAPES, CNPQ, FAPEPI.

References:

[1] de Oliveira, L. H., Trigueiro, P., Souza, J. S. N., de Carvalho, M. S., Osajima, J. A., da Silva-Filho, E. C., Fonseca, M. G. Montmorillonite with essential oils as antimicrobial agents, packaging, repellents, and insecticides: An overview. Colloids and Surfaces B: Biointerfaces, 209, 112186, 2022.

[2] Lima, I. S. D., Ferreira, M. O. G., Barros, E. M. L., Rizzo, M. D. S., Santos, J. D. A., Ribeiro, A. B., Estevinho, L. M. Antibacterial and Healing Effect of Chicha Gum Hydrogel (Sterculia striata) with Nerolidol. International Journal of Molecular Sciences, 24, 2210, 2023.

Formation of nanostructured oxide layer on titanium alloys substrates manufactured by additive manufacturing

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Abstract

Surface modification of Ti implants can improve their performance. It is known that the formation of a nanostructured oxide layer on Ti surface improves its osseointegration [1]. In this work, electrochemical anodization was used to obtaining of nanostructures on Ti-53Nb, with low and high O content, Ti-13Nb-13Zr and Ti-6Al-4V alloys. The Ti substrates were manufactured by powder bed fusion techniques. Two different electrolytes were used for anodizing: organic and aqueous. The nanostructures were characterized by scanning electron microscopy (SEM) and grazing-incidence X-ray diffraction. Nanostructures were obtained on the Ti alloys surface for both electrolytes. The nanostructures presented higher inner diameters but lower thickness when formed using an aqueous solution. The nanostructured oxide layer on the titanium surface did not present a crystalline arrangement. A relatively high O content in the Ti-53Nb substrate caused disorganization in the nanostructured layer. For the Ti alloys substrates with $\alpha+\beta$ microstructure, perpendicular nanostructures to the substrate plane similar to walls were observed, besides perpendicular nanostructures of these walls. The experimental results indicate that the nanostructure growth rate depends on the chemical composition of the crystalline phase.

Acknowledgements

The authors are grateful to the MCTI/SISNANO/INT-CENANO-CNPQ (Process № 442604/2019) and the LNNano (National Nanotechnology Laboratory) at the CNPEM (National Center for Research on Energy and Materials) for the BSE-SEM analysis of the Ti alloys substrates and the SE-SEM analysis of the nanostructures, respectively. E. Sallica-Leva gratefully acknowledges the Brazilian National Research Council (CNPq, Brazil) for granting postdoctoral research fellowships (Grant no. 302115/2021-8).

References

[1] Grimes, C.A., Mor, G.K. TiO2 nanotube arrays: synthesis, properties, and applications. Springer, 2009. ISBN: 978-1-4419-0067-8

Functionalization of titanate nanotubes (NtsTi) and application in antibacterial activity

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Nanomaterials exhibit exciting properties, such as high surface area and chemical stability, showing broad applicability. TiO2-based nanomaterials, such as titanate nanotubes (NtsTi), have been extensively studied in the literature due to their exceptional properties, including drug release, photocatalytic activity, and, most notably, their ability to coat bone implants with biological activities [1]. NtsTi can also be functionalized through the reactivity of OH groups on their surface, including silanization using 3-aminopropyltriethoxysilane (APTEs), which adds an amino-silane group, increasing its dispersibility and antibacterial properties [2]. In this study, we proposed the functionalization of NtsTi with APTEs to evaluate its antibacterial activity against S. aureus and E. coli. The NtsTi were obtained through alkaline hydrothermal synthesis using a Teflon reactor and mixing 1g of TiO2 in 100mL of 10 mol L-1 NaOH heated at 120°C for 24h. Excess alkali was removed, and the solid was washed with 0.1 mol L-1 HCl until pH three and then with deionized water until neutral pH. For functionalization, 150mg of NtsTi was dispersed in 100mL of toluene, added 3.5mL of APTes, and refluxed at 80°C for 8h. The functionalized NtsTi was washed with toluene/ethanol. The material was analyzed by Fourier Transform Infrared Spectroscopy (FTIR), Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), and evaluation of antibacterial properties. The analysis confirmed the formation (NtsTi) with anatase crystalline phases, referring to crystallographic planes (101) and (200), and the functionalization of nanotubes (NTsTi-NH2) with a diameter between 7 and 10 nm. Additionally, both materials showed an inhibitory response to bacterial growth at concentrations of 25 and 50mg L-1; considering the diameters of the inhibition halo, the samples are resistant to bacteria.

[1] I.Neta,Cerâmica 6, 440-450, 2020
[2] W.Zhang, Silício 14 , 6535-6545, 2022

Functionalization of titanate nanotubes with amine groups

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The development of materials on a nanoscale has been driven by advances in technology that can provide materials capable of performing a specific role. As a result, titanate nanotubes (TiNT) are nanomaterials with high biocompatibility, porosity, surface area, and excellent photocatalytic properties. However, the challenge encountered for this material is related to its natural tendency to agglomerate, which can reduce the effectiveness of its properties. Therefore, surface modification with the amino group can contribute to electrostatic interactions, increasing the contact area and facilitating cell adhesion. In this work, titanate nanotubes were functionalized with different concentrations (0.2, 1, and 2 % (v/v)) of 3-aminopropyltriethoxysilane (APTMS) to determine the best functionalization parameter to obtain the best material property. Morphological changes were evaluated by scanning electron microscopy (SEM-FEG), infrared spectroscopy (FT-IR), Raman spectroscopy, and X-ray diffraction (XRD). The results showed that there was no effective functionalization at higher concentrations (samples with 2% and 1%) because silica formation occurred on the material's surface, which was unsatisfactory. However, at a concentration of 0.2%, it was observed that the tubular morphology of the TiNT was preserved after surface modification, and the results of FT-IR, Raman, and XRD showed the presence of functional groups on their surface. Therefore, the parameters used in the sample with 0.2% APTMS were considered the most suitable for this study. Acknowledgement Fapesp process 2020/12507-6.

Glycerol based solvents providing ZnO particles

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The Glycerol-Urea method (GU) is quite excellent for synthesizing ZnO NPs with well-defined size and morphology, and presents great versatility, allowing the control of morphology by using additives [1]. In this study, urea, a key component in the synthesis, was replaced by other isostructural solvents (isopropanol, tetramethylurea, dimethylurea and thiourea) and/or simply bubbling ammonia in glycerol. The aim was to evaluate whether such modifications could bring improvements and gains to the glycerol-based method. For the Glycerol-Isopropanol (GI), the molar ratio of the solvents was varied, reaching at an optimal one for 10:1. The GI ZnO NPs presented spherical morphology and well-defined size and distribution (similar to the GU method). The approaches with Glycerol-Dimethylurea and Glycerol-Tetramethylurea showed ZnO NPs with reduced size, and the Glycerol-Thiourea mixture resulted in a different compound expected: ZnS. Bubbling ammonia into glycerol resulted in ZnO needle-like morphology. The characterizations were carried out with XRD, SEM and MET, and the results were satisfactory, showing that alternatives to urea are useful and can bring synthetic improvements, in addition to obtaining different structures and even a different product, as was the case of ZnO nanoneedles and ZnS.

References:
[1] Brazuna, L. P., Tabuti, T. G., de Paula Silva, A., Tada, D. B., Politi, M. J., Bacani, R., Triboni, E. R. Effect of lithium and sodium ions on the size and morphology of ZnO nanoparticles synthesized by a glycerol-urea route. New Journal of Chemistry, 43(48), 18988-18995, (2019).

Acknowledgements:
Grant nº 021/01509-0, Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP)

GRAPHENE OXIDE ANTIBACTERIAL ACTIVITY FOR MEDICAL DEVICES

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Nanomedicine applies nanotechnology to healthcare, addressing challenges such as bacterial adhesion to medical devices. Antimicrobial nanostructures, including graphene, offer advantages such as low toxicity, overcoming resistance, and reduced cost [1]. This study aimed to obtain graphene oxide (GO) and evaluate its antimicrobial and antibiofilm properties for biomaterial coatings. GO was synthed from graphite using the modified Hummers-Offeman method [2], which involves oxidation and exfoliation. Graphite and GO were analyzed by X-ray diffraction, revealing the presence of graphene oxide sheets. The antimicrobial tests followed the protocol by Trentin et al. (2013) [3]. Chemical composition analysis indicated the introduction of functional groups characteristic of GO during oxidation. Thermogravimetry showed good thermal stability of GO, with minor variations at higher temperatures. Scanning Electron Microscopy (SEM) of GO revealed characteristic sheet structures with roughness. For the antibacterial evaluation of graphene oxide, concentrations ranging from 1.5 mg/mL to 0.1 mg/mL were tested against Staphylococcus epidermidis and Escherichia coli. The lowest concentrations exhibited greater inhibition of E. coli biofilm, with 0.1 mg/mL inhibiting 55.3% and 0.5 mg/mL inhibiting 56.7%. S. epidermidis did not show inhibition. The results suggest that GO has potential for modifying medical device surfaces, demonstrating 55.3% inhibition of E. coli biofilm. Future prospects involve combining graphene oxide with PMMA for antibacterial coating formation.

References

[1] M. Yousefi et al., C, vol. 74. Elsevier Ltd, pp. 568-581, May 01, 2017..
[2] C. R. Minitha, M. Lalitha, Y. L. Jeyachandran, L. Senthilkumar, and R. T. Rajendra Kumar, Mater Chem Phys, vol. 194, pp. 243-252, Jun. 2017.
[3] D. S. Trentin et al., PLoS One, vol. 8, no. 6, Jun. 2013.

Green calcium nanoparticles based on eggshell for dentistry applications

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The proliferation of different pollutants caused by anthropogenic activities has become a significant global concern, as they cause severe health and environmental harm. In this context, the amount of waste disposable by different industries causes a real disorder. Many industries disposable different types of waste, and the search for an economical way to reuse it is the major challenge. Then, the development of new sustainable, low-cost alternative use of industrial waste or co-products using non-classical material is a necessary change [1]. In this context, the agroindustry produced a huge amount of waste that are incorrectly discharged [2]. Considering that, this research performed a green chemical synthesis of calcium nanoparticles using eggshell from agroindustry for dentistry applications. These nanoparticles were synthesized using a heat synthesis using different times (7, 15 and 24 hours). In addition, they are fully characterized by UV Visible, FTIR, XRD spectroscopies, Zeta Potential, and morphology analysis (SEM, DLS). The results showed nanoparticles in the range of 202.27-404.23 nm using DLS for nanoparticles by 15 and 24 hours. The charge using Zeta Potential showed a positive charge, but very close to zero point (2.58 mV), which was line with morphological results obtained by SEM, showing a clustering formation. FTIR showed that nonsignificant changes are perceived in different calcium nanoparticles. In addition, the XRD showed a crystallinity characteristics of calcium oxide. These results showed a great potential for this innovative calcium nanoparticle based on waste from agroindustry for dentistry applications.

Acknowledgment:
The authors thank FAPEMIG (APQ-02565-21), CAPES, CNPq and UFVJM for financial support to the project.

References:
[1] Borsagli FGLM Borsagli A., J Polym Environ, v. 27, p. 1542-1556 (2019).
[2] ZHANG, Minglong et al. Environmental Research, v. 205, p. 112509, 2022.

Growth and Characterization of the (C2H5AgNO2)NO3 crystal

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Silver has been used for medicinal purposes for thousands of years by ancient peoples due to its antibacterial, antifungal, and antitumor properties [1,2]. In this work, the (C2H5AgNO2)NO3 crystal was synthesized by a slow evaporation method. The X ray diffraction (XRD) technique and the Rietveld method, confirms that the material grew in a monoclinic system with P21/a-space group [3]. Thermal properties were analyzed using thermogravimetry (TG), differential thermal analysis (DTA), and differential scanning calorimetry (DSC) techniques. The XRD data collected as a function of temperature revealed that the material undergoes a phase transition at 390 K, transitioning to the triclinic crystal system with the P1-space group. In addition, the vibrational modes were investigated by Fourier transform infrared spectroscopy (FTIR) and assigned using density functional theory (DFT). The findings suggest that the material is promising for applications in antitumor drugs.

Acknowledgments:
We would like to thank CAPES, CNPQ, and FAPEMA for their support in this research.

References:
[1] Banti, C.N. and S.K.J.M. Hadjikakou, Anti-proliferative and anti-tumor activity of silver (I) compounds. 2013. 5(6): p. 569-596.
[2] Liang, X., et al., Recent advances in the medical use of silver complex. European Journal of Medicinal Chemistry, 2018. 157: p. 62-80.
[3] Choudhury, R.R., et al., Ferroelectric glycine silver nitrate: a single-crystal neutron diffraction study. Acta Crystallographica Section B, 2013. 69(6): p. 595-602.

Hybrid System Supported on Bacterial Cellulose for Controlled Release of Bactericide Agents

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A wound is a disturbance of the tissue''s standard shape and function. Skin disruption happens in skin lesions due to physical, thermal, or chemical damage or certain underlying medical or pathological conditions. Gram-positive, sphere-shaped bacteria such as Staphylococcus aureus are a significant health issue due to their multidrug resistance (MDR). Methods for delivering bactericides straight to incisions are being researched. Chemical and photochemical compounds such as safranin and phenothiazines are excellent options for attacking bacteria through chemical and photochemical mechanisms. However, the water solubility and rapid release restrict their effectiveness. In this study, a new hybrid biomaterial was created that combines bacterial cellulose (BC) hydrogel and cellulose nanofibers (CNF) for use as a local drug delivery implant for skin treatments using safranin, which has excellent antimicrobial action against Gram-positive bacteria. CNF increased the safranin load by around two times more than safranin only incubated in BC, indicating a kinetically controlled release mechanism. Bacterial cellulose was also efficient in adsorbing phenothiazine radical cation and its corresponding oligomer, which is promising for application in photodynamic therapy.

Hydroxyapatite deposition on Ti6Al7Nb alloy through electrodeposition and biomimetic method

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This work investigates the deposition of calcium phosphate on the Ti6Al7Nb alloy by two methods: biomimetic and electrodeposition. The Ti6Al7Nb alloy was divided into three groups: one containing only substrate; in the second group, a film of $Al_2O_3$was deposited on the samples with a thickness of 100 nm and in the third, the same film was deposited, but with a thickness of 200 nm. The deposition was performed using the Atomic layer deposition (ALD), which forms thin films that protect the titanium surface [1]. A 5× SBF (simulated body fluid) solution was prepared and the samples were immersed in an incubation period for 7, 14 and 21 days. Another HAp solution was made for electrodeposition. The alloy groups were immersed in the solution and left for a period of 4 hours with heating at 70°C. Raman spectroscopy and X-ray diffraction (XRD) confirmed the presence of HAp and tetracalcium phosphate (TCCP) in the alloy after electrodeposition and SBF processes. The EDS identified the chemical compositions of the apatites in the alloy and the Scanning Electron Microscopy (SEM) showed the crystals formed from the apatites [2]. Both techniques were effective in the deposition of HAp, however, electrodeposition is faster, while SBF lasted up to 21 days for some groups of samples.

Acknowledgements:
LIMAV, UFPI, CNPQ, CAPES.

References:

[1] SPAJIĆ, Ivan *et al.* The effect of surface preparation on properties protective propertiesof $Al_2O_3$and HfO_2thin films deposited on cp-titanium by atomic layer deposition. Electrochimica Acta, v. 366, p. 137431, 2021.

[2] SILVA, André DR; PALLONE, Eliria MJA; LOBO, Anderson O. Modification of surfaces of alumina-zirconia porous ceramics with Sr^{2+}after SBF. Journal of the Australian Ceramic Society, v. 56, n. 2, p. 517-524, 2020.

Improvement of scratch resistance of PEEK by addingHA and ZnO nanoparticles simultaneously for biomedical application

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Human bone tissue has attracted considerable public health concern, due to an increase in the number of bone injuries. Hence, biomaterials are used to repair or replace bones when spontaneous repair may fail [1]. Poly(ether-ether-ketone) (PEEK) is a biomaterial used in orthopedic implants because of its biocompatibility, good tribological properties, and mechanical properties closer to that of human bone [1]. Therefore, this work aims to prepare PEEK nanocomposites by extrusion, with hydroxyapatite (HA), to increase the osseointegration of the matrix, and zinc oxide in spherical (cZnO) and flower (fZnO) morphologies, for tribological reinforcement. The nanoparticles' effect was verified individually, with simple nanocomposites, and simultaneously, with hybrid nanocomposites by mixing HA and ZnO. The specimens were tested by scratching tests in order to analyze their behavior when subjected to pure abrasive wear. The tests were performed in a tribometer using diamond indenters of 200 ・m nominal tip radius to make scratches of 3 mm length, under linearly increasing loading from 1 to 18 N at 0.05 mm/s rate, for 60 s. The residual depth of the scratches was measured by profilometry. The results showed that, between the two morphologies of ZnO nanoparticles, fZnO has a greater capacity to reinforce PEEK, resulting in less scratching depth. HA did not disturb the reinforcement effect of fZnO. Moreover, the scratching of the cZnO-HA hybrid nanocomposite generated less friction, which is bad for the application, as it can cause the loosening of the implant. So, the fZnO-HA hybrid nanocomposite is a promising alternative when considering, for example, application in hip prosthesis stem, as this mixture can provide greater resistance to scratching and greater stem fixation within the bone.

Acknowledgements:

FAPERJ, CAPES and Department of Polymer Engineering (University of Bayreuth).

References:

[1] M.R.Senra, M.F.V.Marques and S.N.Monteiro. Polymers, vol. 15, page 373 (2023).

Improving the mechanical and biological properties of3D Bioprinting oxygen-generating GelMA hydrogels constructions reinforced with electrospun nanofibers

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To date the application of soft hydrogels to bone and cartilage regeneration is limited due to low mechanical modules (fracture and stiffness). To solve this, cryo(bio)printing surges as an alternative to improve the crystallinity of cross-linked hydrogels[1]. At same time, the lack of oxygenated bioprinted hydrogels construction can generate hypoxia inside hydrogelsand promotes cellular apoptosis. Oxygen supply for in vitro cell culture and in vivo 3D bioprinted structures is challenges to improve vascularization inside hydrogels structures. Ifcontrolled, oxygen levels must be maintained in many soft and hard Human tissues to promote the differentiation, viability, and proliferation of cells and in vitro/in vivo tissueformation. Although some technologies employ oxygen-generating materials to add sufficient oxygen levels, the limitations and challenges of current technologies include the lack of controlled, sustained, and prolonged release of oxygen. Herein, we disclose the engineering of oxygen-generating micro/nanoparticles in cells-laden GelMA hydrogels reinforced with nanofibers mats (more than three layers). GelMA displays tunable rheological and mechanical properties after addition of electrospun nanofibers mats (specialafter three layers), for sustained and prolonged oxygen-release. GelMA was cryo (bio)printing at a low temperature -20°C. Three groups were photo-crosslinked at 1, 5, and10 min, when the hydrogel constructions were cryo (bio)printing, by fluorescence microscopic analysis the results showed that highly ordered and parallel channels were evident, indicating a high specific surface area; therefore, smaller pore , lower degradation,and increased stiffness are reported in hydrogels at a lower temperature in comparison withGelMA hydrogels bioprinting room temperature. Our developed strategy demonstrated promising cell response within the high mechanical modulus engineered hydrogels constructions.

Influence of laser modification of the surface of a beta titanium alloy on its fatigue life

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Laser technologies are known to provide significant improvements in the interaction between the substrate of a titanium implant and the bone tissue. The fact is that, as with any surface modification process, the laser can affect the fatigue behavior. This work compared the high cycle fatigue life of a commercial beta type titanium alloy, Ti-15Mo, with polished and Yb:YAG laser modified surfaces. Axial fatigue tests were performed under atmospheric conditions using a stress ratio of 0.1. The results revealed that for the employed process parameters, biologically promising according to the literature [1,2], the fatigue life of the beta type titanium alloy was affected by the laser modification. This follows a behavior similar to that of traditional titanium alloys and therefore requires great caution in the possible use of the laser as a surface modification process.

Acknowledgements:The authors are grateful to The Brazilian National Council for Scientific and Technological Development - CNPq (process 151114/2022-6). Fapesp process 2021/02057-6. A special thanks goes to Prof. Dr. Antonio Carlos Guastaldi for providing the laser surface modification.

References:

[1] Guastaldi, F. P. S. et al. Lower susceptibility of laser-irradiated Ti-15Mo surface to methicillin-resistant Staphylococcus aureus cells adhesion. Materials Research, v. 22, n. 4, 2019.

[2] Santos, M. L. et al. Preparation of laser-modified Ti-15Mo surfaces with multiphase calcium phosphate coatings. Materials Research, v. 23, n. 4, 2020.

Influence of MgO on structural and spectroscopic properties of borophosphate bioactive glasses

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Borophosphate glasses showed to be promising for medical applications, especially in the conformation of glass fibers, scaffolds and bioactive coatings of metallic implants for bone replacements [1,2]. This study aimed to analyze the structural and spectroscopic modifications caused by the addition of magnesium oxide in the system $40B_2O_3$-$25P_2O_5$-(20-x)Na_2O-$15CaF_2$-xMgO (x = 0, 2.5, 5, 7.5 and 10 mol%). The samples were synthed by melt-quenching method, characterized by volumetric density, molar volume, X-ray diffractometry (XRD), differential scanning calorimetry (DSC), Fourier transform infrared spectroscopy (FTIR) and tested in simulated body fluid (SBF) for 28 days to verify bioactivity. The formation of calcium phosphates (CaP) after SBF was investigated via FTIR analysis. XRD diffractograms confirmed the amorphous nature of the glasses. The increase in volumetric density and reduction in molar volume was observed for replacement of Na_2O by MgO. The DSC data showed increase in the glass transition temperature with the decrease in Na_2O/MgO ratio. FTIR spectra before SBF indicated structural changes in the glasses. Deconvoluted absorption bands of FTIR spectra suggest that the increasing of MgO caused depolymerization of vitreous matrix. However, the presence of MgO led to the formation of linear chains between forming species due to the crosslinks made by Mg^{2+} that are more stable than P-O-P, B-O-B and P-O-B linkages. FTIR spectra after SBF showed formation of CaP on all glasses. The results suggest that MgO increases the strength of bonds between the forming species of the samples. Finally, the glasses containing MgO are candidates for the manufacture of bioactive glass fibers.

Acknowledgements:

We are thankful the CAPES, CNPQ, FAPEMA, PPGCM-UFMA and LEOF.

References:

[1] S. J. Watts, R. G. Hill, M. D. O' Donnell, R. V. Law. J. Non. Cryst. Solids 356, 517 (2010).

[2] M. Seshadri, C. Batesttin, I. L. Silva, M. J. V. Bell, V. Anjos. Vib. Spectrosc. 110, 103137 (2020).

Influence of surface charge to the hydroxyapatite deposition on titanium modified by calcium titanate.

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Ceramic materials based on calcium phosphate such as hydroxyapatite (HAp) play an important role in the biomineralization due to its similarity to the bone mineral matrix and, for this reason, they have been used as coatings for implant materials. However, one of the challenges is its low adherence to the metallic biomaterial such as titanium. To ensure a good bonding strength of the HAp, the titanium surface can previously be modified by chemical processes or coated with an intermediate layer [1]. The present work aims to coat titanium with calcium titanate and study the influence of surface charge (zeta potential) of substrates on the hydroxyapatite deposition. The deposition of calcium titanate films on commercially pure titanium (c.p.-Ti) was performed by the sol-gel method associated with dip coating technique, at a linear immersion rate of 5cm/min. It was then hydrolyzed under atmospheric humidity and temperature and finally, calcined at 400°C. The hydroxyapatite was prepared by the sol-gel method starting from $H_3PO_4$and $Ca(NO_3)_2$as alcoholic solution, deposited by immersion at 5cm/min and calcined at 700ºC. The samples were characterized using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersion X-Ray Spectroscopy (EDX) and Surface Zeta Potential measurements. The analysis of zeta potential indicated that both, pure titanium and titanium coated with calcium titanate surfaces favor the adsorption of phosphates first and then the calcium adsorption, explaining the difficulty of apatite deposition from used sol. In addition, by determining isoelectric, it has been shown that the coatings favor the attraction of Ca^{2+}ions to the surface at physiological pH (7.4) and may contribute to the cell growth.

Acknowledgements: CAPES and CNPq.

References:

[1] Ronoh, K. et al. Advances in sustainable grinding of different types of the titanium biomaterials for medical applications: A review. Biomedical Engineering Advances, v. 4, p. 100047, 2022.

Influence of temperature on obtaining apatites by sol-gel method

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Calcium phosphate-based ceramics (apatites) are synthetic materials that have a chemical composition similar to bone tissue, which makes them interesting to use as biomaterials [1]. In this work, the apatite precursor sols at concentrations of 0.7 mol/L and 1.4 mol/L are prepared by dissolving calcium nitrate and phosphorus pentoxide in ethanol. The sols are kept at rest for 24 hours for hydrolysis (gelatinization) under room temperature and humidity. After hydrolysis, the resulting gels are dried at 80°C to remove the organic solvent and then calcined under different temperatures (from 500°C to 1200°C, with increments of 100°C) for 8h [2]. The X-ray diffraction analysis of the samples by the Rietveld method indicates that the samples are composed of monoclinic and hexagonal hydroxyapatite (HAp), the former being predominant, and calcium β-triphosphate (TCP)[3] .For the material started from the sol with a concentration of 0.7 mol/L, the hydroxyapatite fraction increases from 28.8% to 66.5% when the calcination temperature increases from 500°C to 800°C. Above this temperature, the fraction of this phase decreases. With a concentration of 1.4 mol/L, the hydroxyapatite fraction decreases from 88.2% to 19.5% progressively with increasing temperature from 500°C to 1200°C. The micrographs revealed the formation of particulate but porous materials that agglomerate without sintering up to a temperature of 900°C, from where the formation of sintered and dense agglomerates is observed. The results of the EDX analysis showed a Ca/P molar ratio ofaround 1.5 for all materials. The cytotoxicity assay, using pre-osteoblast cells (MC3T3-E1,Subclone 4) by non-direct contact of 24h, showed that cell viability and adhesion did notdiffer from those of the control. A decrease in viability and adhesion was observed for samples prepared at a concentration of 0.7 mol/L and calcined at temperatures above 900°C.

Inhibition of biofilm formation on titanium surfaces by non-thermal atmospheric plasma jet

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The aim of this in vitro study was to evaluate the antimicrobial efficacy of a non-thermal atmospheric plasma jet in inhibiting biofilm formation. Biofilms can cause serious infections and compromise the functioning of implants or injured tissues. The irreversible adhesion of bacteria is the first step of biofilm formation and can be prevented by methods that control and inhibit this adhesion. The atmospheric plasma jet is a promising method, as it is safe, easy to apply in physically complex environments and not prone to microbial resistance [1, 2]. However, there are still a lack of relevant standards for the clinical application of plasma devices, which stimulates the growth of research and studies in the area. In this study, titanium surfaces were exposed to an argon plasma jet and analyzed for their wettability and biofilm formation by Pseudomonas aeruginosa in 24-well plates for 48h. The plasma treatment significantly changed the hydrophilicity and chemistry of the treated titanium surface. The mature biofilms of P. aeruginosa formed on the treated surface showed a reduction of 92.32% (p=0.0001) in the viable cell count (CFU/mL) compared to the untreated control. These results are discussed in light of the reactive nitrogen and oxygen species (RONS) present in the plasma and their influence on the formation of functional groups on the treated surface. In conclusion, our study demonstrated that the argon plasma jet can alter titanium wettability and inhibit biofilm formation.

Acknowledgements:
National Institute of Surface Engineering, CNPq.

References:

[1] Patange, A. D., Simpson, J. C., Curtin, J. F., Burgess, C. M., Cullen, P. J., Tiwari, B. K. (2021). Inactivation efficacy of atmospheric air plasma and airborne acoustic ultrasound against bacterial biofilms. Scientific reports, 11(1), 2346.

[2] Gupta, T. T., Ayan, H. (2019). Application of non-thermal plasma on biofilm: a review. Applied Sciences, 9(17), 3548.

Investigation of multi-element-doped mesoporousbioactive glass by multi-technical spectroscopic approach

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Bioactive glasses (BGs) have been investigated for numerous tissue engineering applications, as multifunctional delivery systems for a variety of therapies, from bioinorganic species to biomolecules (e.g., drugs, growth factors, DNA, contrast agents for diagnostic imaging) [1]. Several studies confirm that bioinorganic species incorporated into the glass composition stimulate important biological activities through ion-mediated therapy and, moreover, are crucial for the formation of the glass network and influence the glassy microstructure and thus the bioactivity of the BGs [1]. The incorporation of biologically active species such as boron, copper, and zinc into bioactive glass compositions has resulted in biomaterials with high bioactivity, interesting antibacterial properties, and stimulation of osteogenesis, angiogenesis, and immunomodulation for tissue regeneration [2]. In this work, we report the synthesis and characterization of multi-elemental glassy compositions derived from the SiO2-CaO-B2O3-ZnO-CuO system. An innovative sol-gel method was used to obtain glasses with high structural homogeneity. The SEM data show that the bioactive glasses have a spherical morphology, while the N2 adsorption-desorption isotherm indicates a mesoporous structure with a high specific surface area for all the studied glasses. The chemical composition of the glasses was confirmed by elemental analysis, yielding values close to nominal. Due to morphological and physicochemical properties, multielement bioactive glasses show great potential as bioactive fillers or drug delivery systems in a variety of applications including bone regeneration and wound healing.

Acknowledgements:

The authors gratefully acknowledge financial support from CAPES and FAPESP.

References:

[1] U. Pantulap, M. Arango-Ospina, A.R. Boccaccini, J Mater Sci: Mater Med vol. 33, 3 (2022).

[2] K. Zheng, W. Niu, B. Lei, A.R. Boccaccini, Acta Biomaterialia vol. 133, 168-186 (2021).

Investigation of the crystallinity of hydroxyapatite thin coatings produced by Nd:YAG (532nm) pulsed laser deposition at room temperature

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Hydroxyapatite (HAP) is a Calcium Phosphate whose crystal structure is similar to bone tissue and is widely used for bone implant coating due to its high biocompatibility and osteoconductivity [1]. Such application demands a crystalline material with the correct stoichiometry of HAP. Heretofore, these conditions have been achieved only with heat treatment, which introduces problems and limitations to the technique. We present and explain the mechanisms to control the crystallinity at room temperature through parameter tuning.

Nd:YAG (532nm) Pulsed Laser Deposition has produced crystalline HAP coatings at room temperature [2], the present work investigates the effects of laser fluence, pulse frequency and pressure on crystallinity.

The effects of the tuning on the crystal structure were determined through Grazing Incidence x-Ray Diffraction, the chemical properties and stoichiometry were measured by x-Ray Photoelectron Spectroscopy.

It is hypothesized that there is a thermal and kinetic energy transfer from the ablated material to the subtract such that room temperature crystallinity is achieved. The kinetic energy of the plasma plume has been determined through Time of Flight type measurements as a function of the different parameters.

The results suggest that the main mechanism is not a kinetic energy transfer, given that it didn’t vary when the pulse frequency was changed, but a difference in crystallinity was observed. Also, as the pressure raised and the kinetic energy lowered, no change in crystallinity was noticeable.

Therefore, another mechanism is responsible for transferring the necessary energy for the crystallization, it is proposed to be thermal. A new experimental setup is being developed for thermal measurements and further investigation.

Acknowledgements

To FAPERJ, CBPF and coworkers of LAPA and LABSURFReferences

[1]W. Habraken, P. Habibovic, et al. Mater. Today 19, 69(2016)

[2]: G.C. Gomes, F.F. Borghi, et al. Surface Coatings Technology, 329, 174(2017)

Investigation of the influence of ZnO addition on the bioactivity of calcium-sodium-borate glass system.

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Borate glasses can be completely converted to hydroxyapatite (HA) and present a higher rate of ion release when immersed in physiological fluids compared to silicates [1]. Metal ions, as Zn2+, can be added to borate glasses to improve their bioactive properties, once this ion has feature of stimulating osteogenesis, wound healing and antibacterial action [2]. In this research, vitreous samples containing ZnO with concentration of 0-10 mol% ((64-x)B_2O_3-2P_2O_5-18 CaO-16Na_2O-xZnO, x= 0, 1, 2.5, 5, 7.5 e 10 mol %)) were synthesized inorder to confirm bioactivity and potential for application in the hard and soft tissues regeneration. The glasses were immersed in Simulated Body Fluid (SBF) for 7, 14, 21 and 28 days to observe the growth of a HA layer on their surfaces in order to attest their bioactivity. The verification of the HA layer was performed by X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FTIR) and daily pH measurement of SBF in which the samples were immersed. XRD, FTIR and cytotoxicity analyzes were also performed before soaking in SBF. Cytotoxicity analysis indicated that all samples are non-toxic, showing no decrease in cellular viability of RAW-47 macrophages. The appearance of characteristic peaks and bands in the DRX analysis and FTIR spectra, respectively, confirmed the formation of HA layer attesting the tissue binding potential of the prepared materials.This study was partly financed by Coordenação de Aperfeiçoamento de Pessoal deNível Superior - Brasil (CAPES) - Finance Code 001. We are thankful to the Brazilian agencies CNPq, CAPES, FINEP and FAPEMA for the financial support.References: [1] P. Balasubramanian, T. Büttner, V.M. Pacheco, A.R. Boccaccini, J. Eur. Ceram. Soc. 38 (2018) 855-869. doi:10.1016/j.jeurceramsoc.2017.11.001.[2] A. Wajda, W.H. Goldmann, R. Detsch, A.R. Boccaccini, J. Non. Cryst. Solids. 511 (2019) 86-99. doi:10.1016/j.jnoncrysol.2018.12.040.

Investigation of TiO_2 Nanotubes Formation on Ti64 Substrates: Effects on Mechanical and Compositional Properties for Biomedical Applications

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TiO_2nanotubes (TNTs) are oxide films grown over Ti substrates via electrochemical anodization in the presence of fluoride ions. TNTs have potential biomedical applications, such as covering implants for bone tissue restoration. Despite the widespread use of Ti-6Al-4V (Ti64) alloys in medical implants due to their bioinert capacity, long-term complications can lead to biological malfunctions. This work aims to investigate the growth, mechanical, and compositional properties of TNTs grown over Ti64 substrates, focusing on biomedical applications. After anodization, characterizations indicated promising results, with well-ordered nanotube formation observed by SEM, and the presence of an initial porous layer on the surface, likely associated with the initial phase of tube formation in organic electrolyte solutions. Mechanical characterization revealed an almost constant elastic modulus for low penetration depth of 24 GPa, indicating the major influence of substrate and densification components on these properties. XPS analysis showed variability in oxide compositions with increasing film depth, possibly attributed to defects in the crystal lattice due to oxide formation. Moreover, the dominance of TiO_2in the films was attributed to the initial surface nanometers, likely correlated with the initial porous layer, which did not form other oxide compositions, possibly due to the smooth formation process during the initial anodization phase that did not introduce defects into the lattice. After annealing at 450ºC for 1.5h, anatase crystal structure was obtained, exhibiting improved properties for biomedical applications.

Acknowledgements:

CEM UFABC and CAPES.

References:

[1] Regonini, D., et. al, Materials Science and Engineering R: Reports, 377-406, 2013.

[2] Li, Zebiao, et al, Materials Science and Engineering: B, 286 (2022).

Laponite-based hydrogel with controlled release of oxygen for applications in skin wounds

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The design of hydrogels that produce oxygen in a controlled manner may offer a unique solution to prevent ischemia-induced death of implanted cells. It indicates oxygen generating hydrogels have the potential to improve tissue engineering strategies that aim to regenerate ischemic tissues. Herein for the first time calcium peroxide (CPO) was laden into Laponite (LAP) hydrogel at different concentrations and evaluated bactericidal effect. The oxygen releasing was measured in hypoxic conditions with control of temperature. After 5h, the concentration decreased to 29.0% for Laponite-CPO (20.0%). However, during the first 5h, Laponite-CPO hydrogels showed increase of oxygen release. The antimicrobial activity is improved significantly with the addition of Laponite to gram-positive and gram-negative microorganisms, improving antibacterial barrier properties. The highest averages in the of the inhibition halos produced by the hydrogels were obtained at concentrations of 5% and 10%. However, it was possible to observe halo reduction, in the highest concentration of CPO. The discs probably adsorbed CaO2, making it difficult to release to the medium. To conclude, Laponite-CPO hydrogels presented antimicrobial activities being a potential candidate for the treatment of infections and consequent helping the healing of skin wounds.

Magnetic enzyme support obtained by sol-gel technique with natural magnetite

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Biocatalysts based on enzymes immobilized on supports have the potential to produce materials with great catalytic efficiency and the reactions can be conducted under mild conditions. In this work, new magnetic support for the immobilization of the lipase enzyme from Thermomyces lanuginosus (TLL) was proposed. A mesoporous and magnetic material was obtained by the sol-gel method in the presence of natural magnetite particles extracted from a Brazilian mine. The synthesis procedure uses eco-friendly concepts since no previous harmful to the environment magnetite synthesis was required. The magnetic xerogel (MX) was modified with gold nanoparticles stabilized by chitosan (MX-Au) and subsequently, TLL enzyme was immobilized on the surface of both materials, to produce MX/TLL and MX-Au/TLL, respectively. The materials were characterized by X-ray diffraction, N2 adsorption-desorption isotherms, thermogravimetric analysis and magnetism. The gold nanoparticles dispersion was characterized by UV-Vis spectroscopy and Transmission Electron Microscopy. The results indicate 5.5 ± 1.6 nm for AuNp diameter, the supports are mesoporous, magnetic and the amount of immobilized enzyme is similar in both materials. The enzymatic activity was evaluated by using the p-nitrophenillpalmitate hydrolysis (colorimetric method). The immobilization parameters were optimized by applying a two-level factorial design which led to the immobilization time of 15 h and pH 5.0. In these conditions, about 66 and 97 % of immobilization yield is obtained for MX/TLL and MX-Au/TLL. Also, the biocatalysts can be reutilized. After 12 cycles, the MX/TLL and MX-Au/TLL biocatalysts preserved 30 and 45% of the initial activity. Moreover, the MX-Au/TLL biocatalyst can be stocked for four months without activity loss. Therefore, the magnetic materials with a planned porous structure and easy synthesis developed in this work have potential application as easy-recoverable and recyclable biocatalysts.

MATRIX VESICLE-EMBEDDED COLLAGEN SCAFFOLDS FOR INVESTIGATING BONE MINERALIZATION

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Bone mineralization is a highly orchestrated and dynamic process that has been studied from a structural, chemical, and biological point-of-view.1The bone tissue can be described by the action of mineralization-competent cells embedded in an extracellular matrix composed mainly by collagen.1,2Those cells release a special class of extracellular vesicles, named matrix vesicles (MVs) which are the responsible for the deposition of an inorganic phase called biological apatite on collagen fibrils.2Thus, the fabrication of 3D-collagen scaffolds embedded with MVs offers alternative strategies to investigate bone mineralization in order to contribute for the development of cell-free therapies. Here, self-assembled 3D-matrices were obtained from the exposure of high concentrated type I collagen solutions to NH3(g) for 24 hours, replicating the microenvironment of the native tissue. MVs were isolated from a pre-osteoblastic (MC3T3) and vascular smooth muscle cell line (MOVAS) cultivated in osteogenic medium. The average mean diameter of both the vesicles was 105 + 19 nm, as determined by nanoparticles tracking analysis. The specific activity of TNAP was 7.5 and 1.2 + 0.6 U.mg-1 for MVs from MCT3T3 and MOVAS respectively, confirming their mineralizing potential. FTIR spectroscopy confirmed the formation of apatite after immersion of the vesicles in synthetic cartilage lymph. Morphological and spectroscopic analysis, revealed the ability of MVs to bind to the collagen scaffolds and propagate the mineral formation from the addition of ATP, simulating the process observed in vivo. Thus, reproducing the organization of collagen fibrils embedded with MVs in vitro is a suitable method to design mimetic models to study (patho)physiological mineralization processes. Acknowledgements: This work was supported by CNPq(200506/2022-6) and FAPESP (2019/25054-2). References:(1)Mansoorifar, A. et al.J. Struct. Biol., 212,107636-107647, 2020. (2) Bottini, M., et al. BBA, 1862, 532-546, 2018.

Mechanical behavior analysis by numerical simulation of titanium alloys for the orthopedic sector

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Abstract:
Orthopedic prostheses combine biocompatibility factors with mechanical properties of the constituent materials. In this perspective, the response induced by Young modulus of the material through the femoral bone and prosthesis are found in the phenomenon of bone remodeling. Studies from the last two decades, controlling alloys phases, denote the management of β phase associated with precipitation of α phase as a promising technique to obtain high mechanical strength with the necessary ductility of biomaterials. Thus, the aim of this study was to evaluate the adequacy of stress distribution of different titanium alloys in view of requests from the orthopedic sector, based on the validations present in computationalsimulations of finite elements to determine possible applications of these alloys in femoral prostheses. From the values obtained with the follow-up of the result regarding the maximum Von Mises tension (σVM) of the healthy femoral bone and the average Von Mises tension (σVM) of the prostheses, it was possible to correlate the stimulus to bone remodeling (ξ), and so the alloy with best performance was Ti-26Nb-4Zr-3Mn.

Keywords: β titanium, femoral prostheses, finite element method.

Introduction

The biomaterials composition demands mechanical resistance in cyclic and repetitive movements, manifested by multiaxial loads [1]. Those requirements are met by titanium alloys, with a young modulus of 110 GPa [2]. The Ti-6Al-4V alloy has the best cost on the market, but it is limited in terms of toxicity, caused by the presence of vanadium and the tendency of aluminum to promote neurological diseases [1]. Another factor that limits the application of this alloy in biomaterials is the progression of conditions resulting from oxidative stress in orthopedic implants [3].

The finite element method tool, illustrated in Figure 1 (l), makes it possible to determine the bone remodeling factor (ξ), which corresponds to the ratio of deformation energy caused by the prosthesis implanted in the femur in relation to the healthy femur, shownin Figure 2 (a), through the mobility of effect meshes by nodes in the three-dimensional geometries of the bodies [1]. This parameter, which is ideal when approximated to the value of “01” for periprosthetic bone nodes, corresponds to a real distribution of stress in healthy bone. In this way, it is possible to obtain the corresponding mobility alloy with betterperformance for femoral prostheses against stress shielding [1]. The purpose of this work was to evaluate the real feasibility of using commercial β-type titanium alloys on hip implants, bya computational tool, based on their mechanical properties.

Experimental Procedure (or Computational Procedure)

The titanium alloys in this work are $\alpha+\beta$ type, including the versatile Ti-6Al-4V alloy, illustrated in Figure 2 (b), conventionally applied in prostheses for THA surgery, in addition to the β-type alloys, namely and shown in Figure 2: Ti-13Nb-13Zr (c), Ti-12Mo-6Zr-2Fe (d), Ti-15Mo (e), Ti-16Nb-10Hf (f), Ti-15Mo-5Zr-3Al(g), Ti-35Nb-7Zr-5Ta (h), Ti-29Nb-4,6Zr-13Ta (i), Ti-26Nb-4Zr-3Mn (j), Ti-35Nb-2Ta-3Zr (k) [1] [2].

Finite element simulations were performed using the ANSYS Student 2022 R1®software, in static structure analysis. The beginning of the modeling of the periprosthetic system started with the use of three-dimensional geometries corresponding to a left femoral bone measuring 416mm in length and an orthopedic prosthesis with a head measuring 36mm in diameter, both widely used and available in open libraries [1]. The manipulation of the software geometries was performed with the attachment of the solid referring to the orthopedic prosthesis, shown in Figure 1(l). Then, the two three-dimensional geometries were unified, in fixation by contact between 3 faces of each geometry, which make up the periprosthetic system, illustrated in Figure 1 (m) and (n). Meshes referring to the junction of discrete nodes were generated and adjusted. In the geometry model section, the lower support was fixed to the bone geometry, at the distal end of the femur, illustrated in Figure 1 (o) and (p), followed by the application of loads analogous to the action of walking [2], which generate a resultant force of 3231.4 N and a reaction of the greater trochanter group of the bone at 1085.2 N.

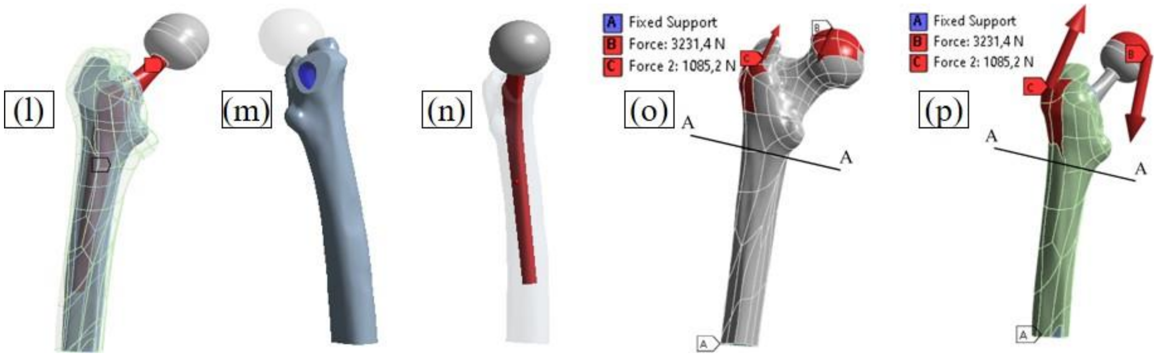

Figure 1: Geometries (l) Fragmentation of hip and periprosthetic system; (m) Medullary system contacts; (n) Femoral prosthesis contact faces. Loads on the greater trochanter and hiphead related to the act of walking. (o) Healthy Bone; (p) Peripsthetic system.

To obtain the bone remodeling factor of each titanium alloy, Image J Software was used to compare the scalar components proportional to the strain energy of each geometry [1].Thus, the Von Mises stress record of the cortical layer of the periprosthetic bone ($VM_{Periprosthetic}$), from the pixel analysis of the A-A section, was divided by the Von Mises stress of the healthy bone ($VM_{Healthy\ Bone}$) of the same A-A section.

Results and Discussion

From the maximum Von Mises stress (σVM) generated in the simulation and the boneremodeling factor ξ, a low or null variation of stresses in the medullary region of the femoral bone in both systems can be observed, shown presented or shown in Figure 2, in the order of7 MPa, illustrated with a blue spectrum. In contrast, there is greater stress dispersion in the cortical region of periprosthetic sections compared to the cortical region of healthy bone, a factor responsible for demonstrating the stress shielding phenomenon, as already expectedand described in the literature [1]. The anteromedial region presented requests to tensions in the average order of 40 MPa. Thus, the Ti-26Nb-4Zr-3Mn alloy, with the lowest elastic modulus (32 GPa), was the material that showed the lowest stress concentration in the critical section A-A, with a maximum of 42.14 MPa and a higher ξ factor of 0.482. It represents the titanium alloy with the best performance, due to its proximity to the elastic modulus of the femoral bone. Conversely, the conventional Ti-6Al-4V alloy showed the highest stress concentration in the critical section A-A, with a maximum of 104.33 MPa and a ξ factor of 0.353, being 36.54% more capable of causing bone remodeling, when compared to theTi-26Nb-4Zr-3Mn alloy.

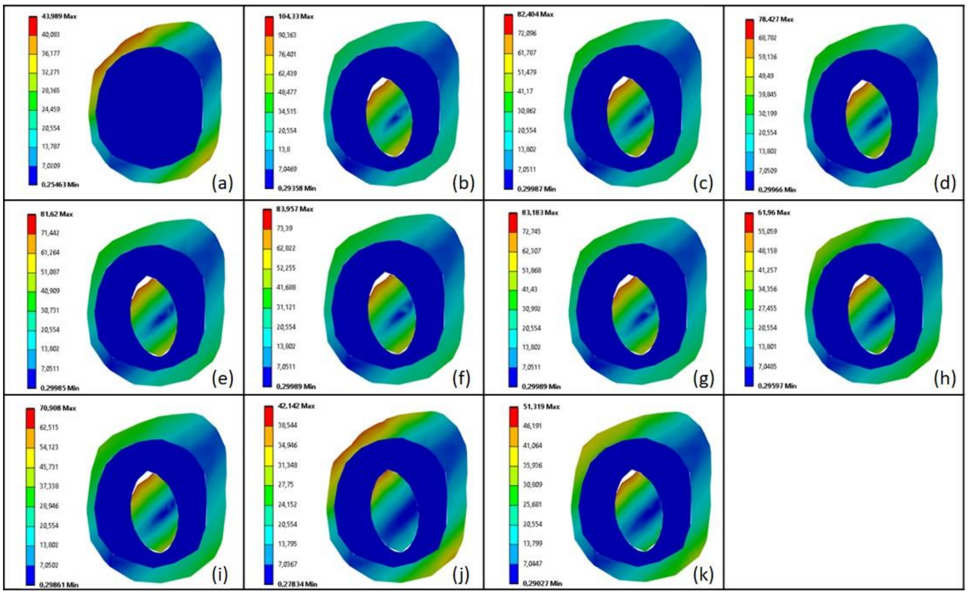

Figure 2: Von Mises stresses distributed in the proximal region of the femur (section A-A).

Conclusions

The computational finite element simulations demarcated the mechanical properties of titanium alloys in relation to periprosthetic bone geometries. The Ti-26Nb-4Zr-3Mn alloy obtained the best performance. The simulations were marked by the low number of simulationnodes and uneven distribution of meshes that limited the accuracy of obtaining the bone remodeling factor. This event should be investigated in future studies.

Acknowledgments

This work was supported by CNPq, members of LCMM-UNIFESP and UFSCar.

References

[1] A. Í. S. ANTONIALLI, C. BOLFARINI. Numerical evaluation of reduction of stress shielding in laser coated hip prostheses. Materials Research, v. 14, n. 3 (2011) 331-334.

[2] H. BOUGHERARA, M. N. BUREAU, L. YAHIA. Bone remodeling in a new biomimeticpolymer-composite hip stem. , v. 92, n. 1, p. Journal of Biomedical Materials Research Part A,v. 92, n. 1, (2010) 164-174.

[3] D. IWANA. Proximal bone remodeling differed between two types of titanium long femoral components after cementless revision arthroplasty. International Orthopedics, v. 32, n.4, (2007) 431- 436.

Mechanical properties and degradation rate of a synthetic bipartite sling modified with a biocompatible and elastomeric midline segment for the correction of stress urinary incontinence

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Urethral suspension surgery combined with the use of synthetic porous tapes (sling) is a worldwide standard and effective procedure for curing stress urinary incontinence (SUI) [1]. However, the force exerted by the sling shortens the urethral canal and causes postoperative urinary retention [2]. In order to avoid the requirement for a second surgical intervention in these patients, a patent was developed in 2010 about a bipartite polypropylene sling interconnected by a small midline segment manufactured from a unspecified biodegradable material that relieves stress over the organ and clears the flow of urine. In this work we evaluated the behavior of a biodegradable poly (glycerol sebacate) elastomer (PGS) used as a midline connection of the bipartite sling and the physicochemical properties of the elastomer. Bipartite slings were made from PGS obtained at different cure times and evaluated for their adhesion strength after in vitro degradation for 7, 14, 21 and 45 days. The PGS elastomer manufactured to be used in slings, were evaluated by infrared spectroscopy (FTIR) and the measurement of swelling percentage and density. The PGS analyzes applied to the slings indicate better crosslinking for 72 hours sample, as well as better performance of mechanical properties over 45 days of degradation. This study pointed out that this polymer obtained by curing for 48 to 72 hours is suitable for use as midline segment of the bipartite slings, although it needs additional in vitro/in vivo studies for specific definition of cure time.

Acknowledgements: This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

References: [1] M. Przudzik, M. Borowik, M. Łesiów, R. Łesiów. International Urology and Nephrology, v. 51, p. 247-252, 2019. [2] E.J. Geller. International journal of women's health, v. 6, p. 829-38, 2014.

Nanocomposites of Titanate-hydroxyapatite applied in regeneration bone

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Strategies for the production of new nanocomposites that promote bone tissue regeneration are important, especially those that potentiate hydroxyapatite osteoinduction in situ [1]. Here, we study the synthesis of nanohydroxyapatite and Titanate (nHAp/TiNT) formulated at different concentrations (1, 2, 3 and 10% dye mass) by means of a wet aqueous chemical reaction. The crystallinity index (CI) calculated by Raman and XRD spectroscopy showed that the values decreased as the tonality concentration increased, which confirmed its addition to the nanocomposite structure. The SEM images showed the presence of shades in the Nanocomposite. The in vitro study, bioactivity assays (SBF 5x), showed the nanocomposites reactivity by increasing the SBF solution's pH after the periods of 7º and14º days and the values calculated for the carbonate content increased with the immersiontime, indicating the presence of carbonated Hap. We also verified the cytotoxicity of the cellline of murine fibroblasts L929, revealing that there was no remarkable cell death in any ofthe concentrations tested. In vivo regenerative activity was performed in models of oophoromized animals (rats) organized into seven groups containing five animals each, in two experimental periods (15 and 30 days), occurring bone regeneration in all groups tested in 30 days. However, the nHAp/TiNT10% group presented statistically higher tissue repair compared to the untreated control group. Thus, the results of this study are promising for numerous applications in bone tissue regeneration.

Ackonowledgements: Financial support from funding agencies CAPES.

References:[1] Oliveira, F. C.; Carvalho, J. O.; Gusmão, S. B. S.; Gonçalves, I. S.; Mendes, l. M. S.; Freitas, S. A. P.; Gusmão, G. O. M.; Viana, B. C.; Marciano, F. R.; Lobo, A. O. (2019). High loads of nano-hydroxyapatite/graphene nanoribbon composites guided bone regeneration using an osteoporotic animal model. International Journal of Nanomedicine

NEW STRATEGY FOR TREATING TUBERCULOSIS: OBTAINING STRUCTURAL, VIBRATIONAL, AND THERMAL STUDIES OF ISONIAZID SALT-TYPE SOLID DISPERSION

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Isoniazid (ISO) is an antibiotic for treating bacterial diseases like tuberculosis. It belongs to the biopharmaceutics classification system class III drug and is unrelated to water solubility problems. However, it presents stability problems in fixed-dose combinations (FDC), commonly used in treating tuberculosis. Solid dispersions of drugs (SDs) such as salts have been an alternative to overcome this problem. Considering this information, this study aimed to obtain and characterize an ISO salt with acetylsalicylic acid (ASA). This salt wasobtained by slow solvent evaporation (SLS) in a molar ratio of 1:3 (drug: drug). Subsequently, the ISO-AAS salt (1:3) was characterized by X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) and Thermogravimetry (TG), Derivative Thermogravimetry (DTG) and Differential Thermal Analysis (DTA). The XRD results showed that the ISO-AAS salt (1:3) exhibited a new crystalline phase and had new crystallographic peaks at 6.0°, 11.5°, 16.3°, 17.9°, 19.2°, 21.2°, and 28.0° 2(θ), indicating an interaction between ISO and AAS. FT-IR spectrum proved the interaction between the two substancesoccurring through the primary amine of ISO (-NH2) and the carboxyl group of AAS, evidenced by vibration at 1500 cm-1 in the ISO-AAS salt spectrum (1:3) referring to the formation of the carboxylate (COO-). Finally, the DTA curve showed an endotherm for melting this salt at 92.6 °C (Tonset). This temperature is lower than the meltingtemperatures of the starting compounds, indicating that ISO-AAS (1:3) will favor an increase in water solubility, bioavailability, and therapeutic efficacy of ISO, supporting the production of more effective drugs in the treatment of tuberculosis.

OBTATION AND SPECTROSCOPIC CHARACTERIZATION OF SOLID DISPERSIONS OF THE ANTIHYPERTENSIVE CANDERSATAN CILEXATIL

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Candesartan cilexetil (CAN) is a medicine used in the treatment of systemic arterial hypertension. It belongs to Class II of the Biopharmaceutical Classification System, presenting low water solubility, contributing to the reduction of its bioavailability and therapeutic efficacy. Obtation solid drug dispersions (DSFs) has become an alternative for improving the physicochemical properties of poorly soluble drugs. This work aimed to obtain and characterize CAN DSFs with tromethamine (TRIS), using slow solvent evaporation (SEL) methanol. After obtaining the CAN-TRIS DSFs, they were characterized by powder X-ray diffraction (XRD) and by Fourier transform infrared spectroscopy (FT-IR). A study of the physical stability of these DSFs as a function of time using the DRXP was also carried out. Difractograms of CAN-TRIS binary mixtures in molar ratios of 1.5:1.0; 1.0:1.0; 1.0:1.5 and 1.0:2.0 were typical of amorphous materials, as they did not show crystallographic peaks. Thus, there is evidence of intermolecular interactions between the starting compounds, resulting in the formation of co-amorphous DSFs. CAN-TRIS co-amorphous (1.5:1.0); (1.0:1.0); (1.0:1.5) and (1.0:2.0) showed good physical stability for up to 168, 365, 308 and 21 days, respectively. The FT-IR spectra of these DSFs confirmed the occurrence of these interactions between the carbonyl and amine groups of CAN and the amino and hydroxyl groups of TRIS. Assuming that amorphous materials are more water soluble than crystalline materials, the CAN-TRIS co-amorphous obtained in this work will contribute to increase the CAN dissolution rates and its bioavailability. Thus, these materials are very attractive for the production of more effective drugs for the treatment of hypertension, as well as for reducing the side effects of this medicine.

Particle size decrease of titanate nanotube by using lyophilization technique

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Materials on a nanoscale have recently received attention due to the search for materials that can be easily manipulated to improve their properties. Among them, titanate nanotubes (TiNT) have received greater attention due to their photocatalytic properties, cost-effectiveness, large surface area, high chemical stability, antimicrobial action, and biocompatibility. However, a challenge encountered is their natural tendency to agglomerate, which reduces their effectiveness. Therefore, this study aimed to analyze the morphological differences between TiNT and lyophilized TiNT. Lyophilization seeks to reduce the agglomeration since the conventional drying process can induce nanoparticle stresses that favor the aggregation. Titanate nanotubes were obtained via hydrothermal synthesis, with one part being dried at 60 °C for 24 hours (sample TiNT) and the other part dispersed in deionized water for 3 minutes using an ultrasonic probe and then frozen at -10 ºC. After that, the sample was placed in a lyophilizer for 3 days at -100 °C under a pressureof 200 μHg (sample TiNT_L). The system’s low pressure promotes solvent sublimation and prevents the particle aggregation. Morphological changes were evaluated by scanning electron microscopy (FEG-SEM), X-ray diffraction (XRD), infrared spectroscopy (FT-IR), and dynamic light scattering (DLS). XRD and FT-IR results indicated no significant alterations in the physicochemical properties of the samples. However, the DLS results showed that the sample TiNT_L exhibited an average particle of 103.2 nm, while the TiNT sample had a particle of 265.5 nm. Furthermore, the FEG-SEM showed that the TiNT_L sample exhibited increased porosity which is a promising outcome for photocatalytic activity and suitable for biological applications.

Acknowledgments: This work was supported by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES)(88887.672990/2022-00)

PCL/Chi and 6-MP's double layer device for potential cancer treatment application

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Mercaptopurine (6-mercaptopurine or 6-MP) treats cancer, particularly leukemia. Common side effects of mercaptopurine include nausea, vomiting, loss of appetite, and increased risk of infection due to immune system suppression. The technology of controlled release of drugs can potentially overpass many problems related to the traditional administration ofactive principles by regulating the rate and spatial localization of the released agent. [1]-[5] Polycaprolactone (PCL) is a biodegradable polyester used in medical applications such as drug delivery and tissue engineering. [6] Chitosan (Chi) is a cationic polysaccharide biocompatible. [7] In this work a doble layer device composed of PCL and Chi/6-MP ‘s were produce using solvent casting and compression molding. The PCL/Chi/6-MP films were characterized by SEM, the results show a multilayer material with modifications on 6-MP's morphology and thickness of approximately 0,25mm. Furthermore, studies will be carried out to evaluate the release of 6-MP.

Acknowledgements: The authors would like to acknowledge NanoSAÚDE, CAPES - Code of Financing 001 and CNPq.

References: [1] S. A. Salim and E. Kamoun, vol. 16, 6937-6955 (2021) [2] K. Schmiegelow and S. N. Nielsen, vol. 36, 503-17 (2014) [3] Y. Zou and D. Mei, vol. 16, 1127-1141 (2021) [4] Y. Zhuang and Y. Su, vol. 15, 1408-1418 (2014) [5] L. Lennard, vol. 43, 329-339 (1992) [6] R. de S. Ribeiro and M. Silva, vol. 3, 423-438 (2022) [7] P. K. Govindappa and D. Joladarashi, vol. 28, 147-154 (2020)

Phenanthroline complexes with Mg^{2+} and Nd^{3+} ions: a comprehensive experimental study combined with computational calculations and cytotoxic assays

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Several scientists and biomaterials engineers have studied phenanthroline coordination compounds involving divalent and trivalent metal ions due to the cytotoxic effect and selectivity that these materials present in various cancer cells [1, 2]. Motivated by this context, here we report the synthesis of two cytotoxic complexes based on 1,10-phenanthroline-magnesium sulfate heptahydrate $[Mg(H_2O)_3(phen)(SO4)]$ and 1,10-phenanthroline-neodymium nitrate hexahydrate $[Nd(Phen)_2(NO_3)_3]$ from the slow evaporation method. Furthermore, the structural, vibrational, thermal, and electronic properties were characterized and discussed. Computational studies based on density functional theory (DFT), Hirshfeld surfaces, and crystal voids supported the experimental analyses. Complementarily, a cytotoxic assay was performed on two cancer cell lines MCF-7 and SiHa, and a healthy cell line GM07492A. The obtained data showed that both complexes crystallized in monoclinic symmetry but with distinct spatial groups. Infrared vibrational spectra presented the main functional groups and exhibited a good correlation to the theoretical DFT results. In addition, considerable thermal stability and electronic dielectric gap were observed for the crystals. From the Hirshfeld surfaces and the lattice voids, it was possible to estimate the intermolecular interactions and unit cell free spaces qualitatively and quantitatively. Finally, the samples showed excellent cytotoxic activity against cancercells and showed higher levels of selectivity for the MCF-7 tumor cell line.

Acknowledgments:

We thank the CAPES, CNPQ, FAPEMA, PPGCM-UFMA, LDRX, and collaborators.

References:

[1] BRUIJNINCX, Pieter CA; SADLER, Peter J. Current opinion in chemical biology, v. 12, p. 197-206, 2008.

[2] MAITY, Basudev et al. Organometallics, v. 29, p. 3632-3641, 2010.

Physicochemical properties of CuZnNi alloys with microbicidal activity

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Copper (Cu) and its alloys have attracted the attention of researchers in recent years due to their natural and microbicidal capacity against various microorganisms for several applications in medicine and as a way to prevent the spread of diseases in environments with high human circulation (hospitals and public transport) [1]. However, the use of pure copper is impaired by the process of oxidation and corrosion that affect the aesthetics and useful life of the material. Thus, the present study evaluated six CuZnNi alloys in order to understand its physicochemical characteristics and microbiological activity against Escherichia coli and Staphylococcus aureus. Cyclic voltammetry, open-circuit potential measurements and potentiodynamic tests were carried out in NaCl 0,5 M in order to determine the corrosion behavior followed by bacterial recovery and plating tests to verify the microbicidal activity using stainless steel 304 as positive control. X-ray diffraction (XRD), optical microscopy, and scanning electron microscopy (SEM) were used to characterize the alloy surface and their corrosion products. The results showed that the Cu68,85Zn26,36Ni4,69 alloy presented the best corrosion behavior (Ecorr= -194 mV and icorr= 1.07 µA.cm-2) compared to other alloys for the same biological activity that promoted full inactivation within 1h of contact with the alloy surface. Furthermore, the addition of Ni promoted a refinement in the dendritic microstructure confirmed by optical microscopy and XRD data confirmed an α-phase crystalline structure. In conclusion, as all alloys obtained the same microbicidal results, the chosen alloy demonstrated better physicochemical properties and could become an alternative for future medical and sanitary applications.

Acknowledgements

The authors thank Termomecanica São Paulo S.A., CNPq, FAPESP, and Embrapii.

References

[1] S.M. Hinsa-Leasure and M.G. Schmidt, Am J Infect Control, 44, 195-203 (2016).

PMMA-silica coatings modified with calcium phosphates and cerium oxide for bioactive protection ofTi6Al4V alloy

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Metal orthopedic and dental implants are widely used for the fixation and replacement of bone tissues. Biomaterials researchers have focused their efforts on developing strategies to minimize implant-associated failures, for example, by surface modification of metal alloys with multifunctional coatings [1]. In this context, poly (methyl methacrylate) (PMMA)-silica hybrid coatings have shown to be very promising for improving the bioactivity, biocompatibility, and corrosion protection of Ti6Al4V alloy [2]. This study focuses on the preparation of multifunctional PMMA-silica coatings modified with hydroxyapatite (HA), β-tricalcium phosphate (β-TCP), and cerium oxide (CeO_2) nanoparticles with bioactive and antibacterial properties. CeO_2 was synthesized by the hydroxide-mediated precipitation method. PMMA-silica hybrids modified with HA, β-TCP, and CeO_2 were obtained by covalently coupling the polymeric phase of methyl methacrylate (MMA) and 3-methacryloxypropyl trimethoxysilane (MPTS) with the silica nanophase, synthesized by sol-gel reactions of tetraethylorthosilicate (TEOS). The 17 μm-thick coatings deposited by immersion on Ti6Al4V are homogeneous and show a strong adhesion (>12 MPa). X-ray diffraction (XRD) and atomic force microscopy showed that the CeO_2 particles have a cerianite structure and sizes in the range of 40–100 nm. The effect of HA, β-TCP, and CeO_2 on the structural properties was evaluated by infrared spectroscopy, XRD, thermal analysis, and contact angle measurements. The corrosion efficiency was studied by electrochemical impedance spectroscopy in a simulated body fluid (SBF) solution. The modified coatings showed an impedance modulus of up to 74 GΩ cm^2, which remains 3 decades higher than the uncoated Ti6A4V alloy after 49 days of exposure to SBF.

Acknowledgments:

This work was supported by CNPq, CAPES, and FAPESP.

References:

[1] Raphel, J., et. al. Biomaterials, 84, 301-314, 2016.

[2] Harb, S.V. et. al. Mater. Sci. Eng. C. 110, 110713, 2020.

Poly(amino alcohol ethers) nanogels embedded with copper ions as an efficient approach for antibacterial therapy.

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The search for new alternative antimicrobial formulations to antibiotics has grown over time in view of increasing resistance to conventional antimicrobials. In this way, the copper ion has shown a broad spectrum antimicrobial action against bacteria, fungi and viruses [1]. In this study, nanogels based on polyetheramine and epoxide [2] were obtained using water as aqueous medium. For this purpose, a polyetheramine (based on propylene oxide PPO) and diepoxy PEG were "click" to obtain the polymeric nanogels. The influence of Cu ions amounton antibacterial properties was evaluated. The nanogels were physicochemical characterized by different techniques such as Dynamic Light Scanning (DLS), zeta potential, Transmission Electron Microscopy (TEM). For biological tests, the minimal inhibitory concentration (MIC) technique was used for the antimicrobial tests and the XTT cell viability assay for the cytotoxicity test. Zeta potential measurements of the pure nanogels revealed ζ-potential of 5±1 mV and from the TEM micrographs was observed spherical morphology of the nanoparticles. The nanogels exhibited antibacterial activity against both Gram-negative and Gram-positive bacteria, which was further enhanced by adding copper ions at low concentrations. Additionally, the nanogels undergo enlargement from 380 to 500 nm with temperature and storage time, while the incorporation of copper does not significantly affect this behavior. These nanogels will be crucial in designing new functional nanomaterials for biomedical applications.

Acknowledgements: The authors are grateful for financial support provided by FAPESP (grants #2021/06552-1, #2022/06507-9, #2022/13408-7 and EMU #2021/14619-9), CAPES (Finance Code 001), and CNPq (grant #307696/2021-9).

References:

[1] Y. Tu, P. Li, J. Sun, J. Jiang, F. Dai, C. Li, Y. Wu, L. Chen, G. Shi, Y. Tan, H. Fang, Adv. Funct. Mater. 31 (2021).

[2] X. Yu, F. Cheng, W. He, Colloids Surfaces B Biointerfaces. 207., 112009 (2021).

Polymer-Based Injectable Nanocomposites Containing Silica Particles for Targeted Delivery of Hydrophilic and Hydrophobic Drugs

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Despite all the advances in the pharmaceutical area, there is still some problems to be overcome, such as the rapid degradation of amino acids and proteins after administration and the controlled release of hydrophobic drugs due to their low solubility, which results in low bioavailability of these compounds [1]. Thus, this study focused on developing localized drug delivery systems for protection and improved efficacy of drugs, using thermoresponsive polymers that undergo a sol-gel transition at physiological temperature. Nanocomposites based on poly(N-vinylcaprolactam) and silica particles were synthed as drug carriers for hydrophobic and hydrophilic drugs [2]. Rheological analysis showed that the nanocomposites can be locally injected to target sites, and the addition of silica nanoparticles minimized premature material dissolution and rapid drug release. The synthed polymers sustained a controlled release of drugs for 7 days under physiological conditions. Kinetic analysis showed that the release of naringin is controlled by diffusion, while the release of doxorubicin is controlled by anomalous transport. A preliminary in vitro study showed negligible toxic effects on healthy cells and better efficient inhibition of carcinoma cells. The study shows that it is possible to use injectable PNVCL-based thermosensitive nanocomposites with silica nanospheres for both hydrophilic and hydrophobic drug delivery with high control and in a minimally invasive manner, which could potentially be applied in future pharmaceutical applications.

Acknowledgements:
The authors would like to thank the financial support of São Paulo Research Foundation (FAPESP) grants #2018/12871-0, #2022/10016-0, #2013/25663-2, #2013/07296-2.

References:
[1] E. Larrañeta, S. Stewart, M. Ervine, R. Al-Kasasbeh, R. F. Donnelly, Journal of Functional Biomaterials 2018, 9.
[2] Lucas S. Ribeiro, Renata L. Sala, Thaiane A. Robeldo, Ricardo C. Borra, Emerson R. Camargo, Langmuir 2023 39 (6), 2380-2388.

Polymeric film based on a PVA/poloxamer blend containing pomegranate peel extract as an alternative dosage form for treating topical candidiasis

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Candidiasis is a highly prevalent fungal infection caused by different species of the *Candida*fungus. The existence of multiresistant *Candida*strains is a public health problem [1]. Extracts from different parts of pomegranate containing ellagic acid and punicalagin have shown antifungal properties [2]. A crude dry extract of pomegranate peel (PPE) containing the bioactive compounds of interest identified by the HPLC method was used in the preparation of polymeric films for topical administration, aiming to obtain na alternative therapy to treat candidiasis. Films based on mixtures between poly(vinyl alcohol) (PVA) (Mw = 13,000 to 23,000 Da; hydrolysis = 87 to 89%) at 25% w/w and poly(ethylene glycol) triblock copolymer (P407; Mw = 12,600 Da) at 20% w/w were prepared by using the solvent casting technique, with different proportions between PVA and P407 (70:20, 60:30 and 50:40 v/v). A hydroalcoholic solution was used as the vehicle, with glycerin as the plasticizer and the PPE was added at 2.5% w/w in the blends. Polymeric films containing PPE were amber-yellow in color, shiny, smooth and without domain formation. The films were easy to remove from the molds and skin, maintaining the integrity during removal. All films demonstrated antimicrobial efficacy on strains of *Candida albicans*, *C. glabrata*, *C. krusei*and *C. tropicali*. The drying times of the films were around 1 h and 15 min (molds) and less than 30 min (skin). Average weight values varied between 0.034 (± 0.011) and 0.095 g (± 0.019); thickness varied between 0.090 (± 0.030) and 0.212mm (± 0.037); and residual moisture from 9.34% (± 0.002) to 12.46% (± 0.022). Physicochemical characterization tests of the polymeric films with and without PPE are in progress.

Acknowledgements: FAPES (Edital 03/2021; TO 427/2021).

References:

[1] BOHNER, F.; GACSER, A.; TOTH, R. Current Tropical Medicine Reports, v. 8, n. 2, p. 59-68, 2021.

[2] DE PAULA, G. A. et al. Natural Product Research, v. 37, n. 4, p. 603-607, 2023.

Polymeric scaffold functionalized with alginate and concavalin a to promote three-dimensional cell culture

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Tissue engineering in association with biomedical science has become an exponent for the use of biocompatible nanostructures in the repair of damaged tissues. The concanavalin A (ConA) lectin is a protein isolated from Canavalia ensiformis with the ability to bind to glycoconjugates, such as those present on cell surfaces. The objective of this work was the development of scaffolds based on polyvinyl alcohol functionalized with sodium alginate and ConA as a favorable environment for cell growth and multiplication. Nanofibers were prepared by formulating PVA and alginate homogenized in deionized. The formulation is inserted into a syringe, electrospinning occurs using a voltage of 24kV. The nanofibers obtained were activated by the coupling agents, aiming at stability in the immobilization of the ConA lectin. Electrochemical and optical techniques were used to evaluate the nanofibers, demonstrating the immobilization of the lectin on the polymeric surface, thus providing cell adhesion factors. The scaffold presents itself as an innovative alternative of three-dimensional material for use in the growth and proliferation of cells, with promising application as supports for skin grafts.

Acknowledgments: Universidade Federal de Pernambuco e CNPq.

References:

[1] M. Hernández, M.A. Álvarez-Pérez, J. Genesca, K.K. Gómez, A. Covelo, Evaluation of the biocompatibility of a PVA/SA scaffold with a human gingival fibroblast (HGF) by using electrochemical impedance spectroscopy, Bioelectrochemistry. 131 (2020).

[2] A.S. Fragoso, M.B. Silva, C.P. De Melo, J.L.A. Aguiar, C.G. Rodrigues, P.L. De Medeiros, J.F. Branco Junior, C.A.S. Andrade, M.D.L. Oliveira, Dielectric study of the adhesion of mesenchymal stem cells from human umbilical cord on a sugarcane biopolymer, J. Mater. Sci. Mater. Med. 25 (2014) 229-237.

Polypropylene fiber as reinforcement for dental pin: Thermogravimetric evaluation.

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Teeth can be severely damaged and in cases of short dental crown it is necessary a pin as support. Nowadays, non-metallic prefabricated polymer composites pins emerged within the dental market [1]. The low cost, improved strength and modulus are the main reasons for their selection [2,3]. The aim of this research was the replacement of inorganic fiber by polypropylene fiber, PP, viewing application as dental pin. The thermogravimetric analysis (TGA) of the samples was conducted in TA equipment model Q500, 30–700 °C, 10 °C/min, under nitrogen. The reference pin curve showed onset temperature (Tonset) around 275 °C. Three stages of mass loss were detected: the first one below 100 °C and decays at 328 °Cand 410 °C. The polymeric matrix represented around 29% and the residue about 70% (inorganic reinforcement). TGA curve of pin containing PP fiber presented the maximumdegradation temperature at 160 °C and 250 °C, representing about 15% mass loss. A thirdstage occurred around 358°C representing around 75% of mass loos. The residue (around10%) was considered as carbonaceous material due to incomplete burning. Infrared spectroscopy and X-ray fluorescence analyses of the reference pin indicated that polymericmatrix was based on epoxy resin reinforced with glass and zirconia fibers. Acknowledgements: UFRJ and Professor João Luiz Portella Duarte (co- advisor).References:[1] Goracci, C., Ferrari, M. Current perspectives on post systems: a literature review. Aust Dent J; 2011; 56:77-83.[2] Minoo N., Shirvanimoghaddam K. Functionally graded materials: A review of fabrication and properties. Appl. Mater. Today.2016; 5:223-245. [3] Schwarzer E., Holtzhausen S., Scheithauer U., Ortmann C., OberbachT., Moritz T., Michaelis A. Process development for additive manufacturing of functionallygraded alumina toughened zirconia components intended for medical implant application. J.Eur. Ceram. Soc. 2019; 39:522-530. doi: 10.1016/j.jeurceramsoc.2018.09.003.

Possible application of hexagonal boron nitride asglucose biosensor using Raman spectroscopy

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The improved technology for glucose (GL) detection will produce an increase in the quality life of millions of diabetics and pre-diabetics around the world. Numerous studies have been undertaken in recent years for the development of in vivo, minimally invasive, biologically compatible real-time quantitative GL sensors. In this sense, the development of biosensors with high sensitivity and low detection limits provides a new direction for medical and personal care. Two-dimensional materials such as graphene and hexagonal boron nitride (h-BN) are being used as biosensors due to their excellent detection performance [1, 2]. Several optical techniques have been used for GL detection, such as: infrared absorption, laser polarimetry, fluorescence modification of dyes. Most of these techniques are not molecule specific and can produce similar results with structurally similar molecules. However, Raman spectroscopy technique is unique, demonstrating the specific vibrational properties of each molecule. According to this, the objective of this work is to verify the possibility of using bilayers (2L), few layers (FL), multilayers (ML) and h-BN bulk as a GL biosensor using the Raman spectroscopy technique. For this purpose, the 2L, FL, ML and h-BN bulk were deposited on SiO2 substrates and subsequently characterized by Raman spectroscopy. Then a drop of commercial GL was placed on the surface of h-BN samples. Subsequently, Raman measurements, in the presence of the GL molecule were carried out in the same regions of the 2L, FL, ML and h-BN bulk samples, with the aim of comparing the Raman spectra before and after the GL deposition. Results of this comparison show that 2L are more sensitive to the GL molecule than FL and bulk and could be used as GL biosensors.

Acknowledgements:
Ao CNPq e a Unipampa

References

[1] A. Ahmadianyazdi, J. Xu and V. Berry, NanoResearch 15(10), 8697-8704 (2022).
[2] N. Basu, M. S. Bharathi, M. Sharma and V. R. Soma, Nanomaterials 11, 622 (2021).

Possible molds for a new brachytherapy source forspinal and intracranial cancer

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Central Nervous System (CNS) cancer is the 18th most incident cancer in the world, according to the World Health Organization (WHO), and in 2020, in Brazil, it represented 2.5% of all new cancer deaths [1]. Brachytherapy is a type of radiotherapy that positions the radioactive source close to (or in contact with) the tumor. Thus, the targeted region receives most of the dose, protecting the healthy tissues adjacent to the tumor. A promising radioactive source for use in brachytherapy for the treatment of CNS cancer is phosphorus-32 in a polymeric substrate. This source has been prominent as a minimally invasive treatment for craniopharyngiomas and in the treatment of metastatic bone diseases in general [2]. To produce such a source, it is necessary to search for possible molds that can conform the polymeric material into the desired format. Several tests were carried out to determine the best mold for the radioactive source manufacture. Different materials were tested to observe if the mold material would react with the source's components, silicone rubber mixed with orthophosphoric acid. In order to be considered a possible mold, the mixture should cure; if the cure did not concretize, the mold would be descarted. It was concluded that polystyrene (PS) obtained the best result, due to the ease of unmold after the resin curing process.

Acknowledgements:

Acknowledgment to the agencies FAPESP 2017/50332-0, CNPQ 309061/2019-9, CAPES 88887.689556/2022-00 and AIEA BRA-6062, for project funding and scholarship.

References:

[1] H. Sung, J. Ferlay, R.L. Siegel, M. Laversanne, I. Soerjomataram, A. Jemal, F. Bray. CA Cancer J Clin, vol. 71(3), pp. 209-249, 2021.

[2] O.I. Solodyannikova 1, V.V. Danilenko 1, G. G. Sukach. Problems of Radiation Medicine and Radiobiology, vol. 26, pp. 562-572, 2021.

Precursor influence in the synthesis of α-wollastonite: a comparative study of using silicilic acid and tetraetilortossilicate (TEOS)

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Wollastonite is a material of great importance in the synthesis of biocompatible prostheses [1] and other medical and dental applications due to its ability to induce the formation of new bone tissue. Variations in the methods and nature of the precursors used in its synthesis can result in materials with different parameters, such as pore and mechanical properties, as well as the rate and products of their degradation, which can influence the biological response of the material [1]. In this study, we investigated different silica sources, such as TEOS (tetraethylorthosilicate) and silicic acid, to assess possible changes in the synthesis of α-wollastonite. The samples were prepared using a sol-gel method, making gradual adjustments to optimize the use of energy and reagents while achieving the desired quality of the final product. Physical and chemical characterization of the powders was carried out using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy, and scanning electron microscopy (SEM). The results indicated that structurally similar and microporous materials were produced, with silicic acid being the most cost-effective precursor.

Acknowledgments:
This study was financed in part by the São Paulo Research Foundation (FAPESP), grants #2023/06336-2 and #2022/07094-3, and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001 (88887.672990/2022-00)

References:

[1] ZENEBE, C. G. A Review on the Role of Wollastonite Biomaterial in Bone Tissue Engineering. Hindawi, BioMed Research International, 2022.

PREPARATION AND CHARACTERIZATION OF NEW SOLID DISPERSSIONS OF ISONIAZID TO INCREASEITS HYDROSOLUBILITY

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Isoniazid (ISO) drug used in the treatment of tuberculosis, belonging to Class III of the biopharmaceutical classification system (BCS), with good water solubility and low membrane permeability, contributing to the reduction of its bioavailability and therapeutic efficacy. Obtaining solid drug dispersions (SDDs) has been a strategy to improve the physicochemical properties of drugs, such as water solubility [1-3]. This study aimed to obtain and characterize new ISO SDDs from the interaction with malic acid (AML), using slow solvent evaporation (SSE) methanol. After obtaining the samples, they were characterized by powder X-ray diffraction (PXRD); and by simultaneous thermogravimetry, derivative thermogravimetry, differential thermal analysis (TG/DTG-DTA). The physical stability of these SDDs was performed by PXRD, as a function of time. The results obtained by PXRD of ISO-AML binary mixtures in molar ratios of 1:2 and 1:3 confirmed the amorphous nature of these new materials, as well as the occurrence of intermolecular interactions between ISO and AML, resulting in the formation of SDDs from co-amorphous type. The physical stability of these co-amorphous was investigated, and the co-amorphous of ISO-AML (1:2) showed stability for up to 49 days. While the co-amorphous of ISO-AML (1:3) was stable for up to 105 days. The TG/DTG results of the co-amorphous showed that these materials showed good thermal stability up to 135°C. The DTA curve of these solid dispersions did not show the occurrence of thermal events related to melting, confirming the amorphous nature of these materials. Assuming that amorphous materials are more water soluble than crystalline materials, the co-amorphous of ISO-AML (1:2) and (1:3) will contribute to increase ISO dissolution rates and its bioavailability. Thus, these materials are very attractive for the production of more effective drugs for the treatment of tuberculosis, as well as for reducing the side effects of this drug.

Printability of Carboxymethylcellulose and Laponite Hydrogels Using by Extrusion-Based Printer

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Hydrogels based on Carboxymethylcellulose (CMC) and Laponite have shown promise as printable inks for tissue engineering applications and drug delivery [1,2,3]. In this study, we investigated the printability of CMC and Laponite hydrogels using an extrusion-based printer. We evaluated the effect of various printing parameters, such as printing speed, multiplier extrusion factor, nozzle diameter, and material concentration, on the printability and rheological properties of the printed structures. We used three different printing speeds (5,7 and 10 mm.s-1), three multiplier extrusion factors (100,125 and 150), and two CMC/Laponite concentrations (1CMC/2LAP and 1CMC/3LAP).

The 1CMC/3Laponite ink exhibited a pronounced pseudoplastic behavior with filament formation during printing and good printability using an Extrusion Factor of 100 and a Printing speed of 5 mm. s-1. On the contrary, the 1CMC/2Laponite ink did not exhibit good printability in most printing parameters studied.

Therefore, we demonstrated that studying printing parameters, printability, and rheological properties could provide insights regarding determining a suitable ink for use in the three-dimensional printing processes of hydrogels.

References:

1. Ahlfeld T, Cidonio G, Kilian D, et al. Development of a clay based bioink for 3D cell printing for skeletal application. Biofabrication. 2017;9(3). doi:10.1088/1758-5090/aa7e96.
2. Habib A, Khoda B. Development of clay based novel hybrid bio-ink for 3D bio-printing process. J Manuf Process. 2019;38 (December 2018):76-87. doi: 10.1016/j.jmapro.2018.12.034.
3. Habib A, Sathish V, Mallik S, Khoda B. 3D printability of alginate-carboxymethyl cellulose hydrogel. Materials (Basel). 2018;11(3). doi:10.3390/ma11030454.

Probing covalent bond between Rhodamine B andorganic polymers

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Polymeric nanoparticles (NP) are promising drug carriers for various diseases. Covalent bonds between polymers and fluorescent dyes can be used to prepare photoactive materials to detect and identify transporters in biological media. Proving that the covalent bond between the polymer and the dye is important to ensure that the observed fluorescence signal identifies the location of the NPs and therefore the target organic structure. However, some techniques are not always sensitive enough to detect these bonds [1]. The photophysical properties of Rhodamine B (RodB) in solution are affected by the chemical environment. In a protic medium, RodB is in the zwitterionic red form, whereas in aprotic solvents the solution is colorless, related to a lactonic specie [2]. The objective of this investigation was to explore this particular behavior of RodB to characterize the formation of the covalent bond between the polymer poly(ε-caprolactone) (PCL) and RodB. This study showed that the isomeric equilibrium associated with the molecular RodB did not occur, as the esterification of the RodB carboxylic acid prevented the formation of the lactonic form. Despite the very few amounts of RodB used to prepare the doped polymer, the proposed methodology showed to be solid to prove the covalent bond between polymer and fluorophore.

Acknowledgments:
Foundation for Science and Technology (FCT), Unit on Applied Molecular Biosciences (UCIBIO), Associate Laboratory Institute for Health and Bioeconomy (i4HB), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) and Laboratory of Molecular Catalysis (LAMOCA - UFRGS).

References:
[1] F. S. Poletto, L. A. Fiel, M. V. Lopes, G. Schaab, A. Gomes, S. S. Guterres, B. Rossi-Bergmann and A. R. Pohlmann, J. Colloid Sci. Biotechnol. 1, 89 (2012)
[2] I. Rosenthal, P. Peretz, and K. A. Muszkat, J. Phys. Chem. 83, 350 (1979)

Production and Characterization of Membranes Basedon Poly-L-lactic Acid Incorporated with Ketoprofen

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Nonsteroidal anti-inflammatory drugs, such as ketoprofen (KET), are commonly prescribed for pain and inflammation treatment (WONGRAKPANICH et al., 2018). However, the possibility of processing membranes with controlled drug release is a challenge in drug delivery (MORIE et al., 2016). Poly-L-lactic acid (PLLA) is a bioabsorbablepolymer commonly used in bone applications in form of membranes (ZAN et al., 2022). Theobjective of this study is to produce PLLA membranes incorporated with 15% (w/w) KETthrough the rotary jet spinning (RJS) process, and to characterize their properties and suitability for tissue engineering (TE) in bone regeneration. The membranes were fabricatedinto fibers and were tested to evaluate the processing and surface morphology, to determinethe chemical functional groups, thermal properties, wettability, absorption and, drug release in a simulated corporeal environment. The morphological results demonstrated thatthe RJS process successfully produced micrometric scale fibers. Chemical analysis revealedthe presence of characteristic functional groups of both PLLA and KET, without any toxic solvent residues. Thermogravimetric analysis revealed that the membranes exhibited thermal stability at human body temperature. The wettability test demonstrated that PLLAmembranes incorporated with 15% KET are hydrophobic. The mass loss test indicated thatan increase in the amount of KET incorporated into PLLA resulted in an accelerated processof mass loss. Additionally, the drug release test confirmed that PLLA membranes with 15%KET were capable of releasing the drug. In conclusion, the produced membrane shows promising potential for applications in bone tissue repair, owing to its anti-inflammatory properties.

Acknowledgements:
We would like to thank the financial support by FAEPEX/UNICAMP #2614/22, CNPq, grant #406514/2021-6 and, the support by CTI' s Open Labs - Multiple Users and Shared Facilities, CTI Renato Archer, research instit

Production of customized bioabsorbable scaffolds by additive manufacturing for bone tissue repair

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Bone tissue engineering aims to treat injuries using 3D matrices (scaffolds), which repair the damaged tissue. As bone is the second most transplanted tissue after blood [1], the study of additive manufacturing for bone application is important due to the production of bioactive scaffolds customized to the specificity of each patient''s injury. Thus, this research produced scaffolds for use in bone tissue engineering using material extrusion-based additive manufacturing technology (Allevi 2, USA). The matrices are composed of poly(lactic-co-glycolic) acid (PLGA), which is a bioabsorbable polymer [2], and hydroxyapatite (HA) nanoparticles, which have osteoconductive properties [3]. The structures had different pore s (450 µm and 900 µm) and 10% w/w HA added (PLGA-HA). Scanning electron microscopy (SEM) results showed that the pore s of the PLGA-HA scaffolds were equivalent to the proposed. Fourier-transform infrared spectroscopy (FT-IR) revealed the characteristic peaks of PLGA and HA, and no peaks corresponding to the solvent used. Thermogravimetric analysis showed that the concentration of HA was added accordingly. Finally, the scaffolds presented osteoconductive properties in vitro. Thus, the scaffolds proposed in this study were successfully processed and have a great potential to promote osseointegration for bone applications.

Acknowledgements

We would like to thank the funding agencies that supported this research, the São Paulo Research Foundation (FAPESP), grant #2020/07923-0, #2020/14679-9, and #2022/00833-1; National Council of Scientific and Technological Development (CNPq) and PIBIC/Unicamp. This research was also supported by TI' s Open Labs - Multiple Users and Shared Facilities, CTI Renato Archer, research institution.

References:

[1] J. DONG et al., Microsystem Technologies, p. 1-15, 2019.

[2] H. CUI et al., Advanced healthcare materials, v. 6, n. 1, p. 1601118, 2017.

[3] D. ZHAO et al., Bioactive materials, v. 6, n. 2, p. 346-360, 2021.

Properties of solubilized β Ti-12Mo-25Nb alloy for biomedical application

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Ti alloys have been studied for biomedical application due to their corrosion resistance, biocompatibility, and better association between their mechanical properties [1-3]. The objective of this work was to analyze the properties and microstructure of treated Ti-12Mo-25Nb alloy. Ti alloys have been studied for biomedical application due to their corrosion resistance, biocompatibility, and better association between their mechanical properties [1-3]. The objective of this work was to analyze the properties and microstructure of treated Ti-12Mo-25Nb alloy. This alloy was produced by arc melting with non-consumable tungsten electrode in controlled atmosphere of argon. The alloy was treated at 950 °C by 1hand water quenching. The material was characterized by X-ray diffraction and optical microscopy. The mechanical characterization was realized by Vickers hardness and Young' smodulus by was obtained by the ultrasound technique. The results of the microstructural characterization showed only the presence of the β-Ti phase. Vickers hardness equal to 210HV and Young' s modulus 74 GPa. With the results obtained, it can be observed that theTi-12Mo-25Nb alloy presents potential for biomedical application.

Acknowledgments
This work was supported by CNPq, CAPES and FAPERJ.

References

[1] Chapala P, Kumar PS, Joardar J, Bhandari V, Acharyya SG. Effect of alloying elements on the microstructure, coefficient of friction, in-vitro corrosion and antibacterial nature of selected Ti-Nb alloys. Appl. Surf. Sci (2019), 617-623.

[2] Xu JL, Tao SC, Bao LZ, Luo JM, Zheng YF. Effects of Mo contents on the microstructure, properties and cytocompatibility of the microwave sintered porous Ti-Mo alloys. Mater Sci Eng C 97 (2019), p. 156-165,.

[3] Chui, P.; Jing, R., Zhang, F.; Li, J.; Feng, T. Mechanical properties and corrosion behavior of β-type Ti-Zr-Nb-Mo alloys for biomedical application. J. Alloys Compd, 842 (2020).

Raman, FT-IR, and DFT studies of *aqua-(glutaminato)-(1,10-phenanthroline)-copper nitrate monohydrate* coordination complex

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An aqua-(glutaminato)-(1,10-phenanthroline)-copper nitrate monohydrate complex was synthesized and its vibrational properties were evaluated by Fourier-transform infrared (FT-IR) and Raman spectroscopy. In addition, quantum chemistry calculations were performed to obtain an understanding of the relationship between the structure and reactivity of metallic complexes. The optimization of the geometry of the titled complex and the vibrational frequencies were performed using the functional PBE1PBE together with the set of basis functions 6-311++G(d,p) for C, O, H, and N atoms, and the Stuttgart-Dresden basis set for copper (Cu) atom, which includes effective core potentials for the relativistic treatment of core electrons and double zeta polarized basis for valence electrons. Chemical, electronic and vibrational properties were properly investigated through experimental and theoretical approaches, including a solvation effects study based on the polarizable continuum model, using methanol and water as solvents. Thereby, the results of this study indicate the potential and relevance of using the density functional theory in the chemistry of organic and metallic compounds, opening a way for advances in understanding the reactivity, structure, and vibrational proprieties of coordination complexes. Finally, the vibrational calculations showed good accordance with experimental Raman and IR results.

Role of aspartic acid in the synthesis of spherical vaterite: study of the crystallization mechanisms in aqueous medium using cryo-transmission electron microscopy techniques

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Vaterite, is the least stable phase of calcium carbonate ($CaCO_3$). The phase vaterite mainly exists in biological systems, rather than in nature [1]. However, some amino acids could stabilize the vaterite phase. Vaterite is expected to have potential applications for biomedical areas as preservative containers for growth factors and encapsulating carriers for drugs because of its high solubility, high specific surface area and high dispersion. Aspartic acid is amino acids that could control the nucleation and the growth of crystallization on increasing the rate of nucleation and delaying the growth of crystal. However, the role of amino acids on the crystallization behavior is still difficult to understand, and the mineralization mechanism of vaterite does not have a complete, reasonable explanation. In this work, we present a sequential analysis of the different steps of $CaCO_3$ synthesis with aspartic acid in aqueous medium. The systematic study of growth mechanisms in the maturation phase of the synthesis was performed using the technique of Cryo -Transmission Electron Microscopy (Cryo-TEM) using the modes of HRTEM, SAED, EDS and EELS. This technique allows preservation of 3 µL of the specimen by vitrification at different stages of the synthesis process, preventing their modification during drying. For the synthesis of $CaCO_3$ we use $CaCl_2.2H_2O$, Na_2CO_3 and aspartic acid (0.1M) what were obtained from Sigma Aldrich. Aqueous solutions of the reactants was made using milli-Q water. We also analyzed the dry sample of the synthesis with other techniques that that need large amounts of material, such as XRD, SEM, FTIR and TGA. The experimental results indicate that the addition of aspartic acid inhibited the growth of calcite, presenting by XRDa 90% of the vaterite phase and SEM clearly shows nanostructures with diameters of micrometers formed by nanoparticles with diameters of 30 nm.

[1] Rodriguez-Blanco, J. D. at. al., Crystal Growth Design. v12 P. 842-853 (2011)

Siloxane-p(HEMA) hybrid coating applied on Ti-6Al-4V alloy as a promising alternative for biomedical applications

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Organic-inorganic hybrids using poly(2-hydroxyethyl methacrylate) p(HEMA) as bioactive coatings for Ti-6Al-4V alloy have been the subject of much research [1]. However, the study of corrosion resistance and biocompatibility simultaneously of this material is limited. Therefore, the present work aimed to evaluate the structural and surface behavior, corrosion resistance and bioactivity of the Siloxane-p(HEMA) hybrid coating deposited on Ti-6Al-4V alloy. The preparation of the hybrid prepared by the sol-gel method occurred in three stages. The first consisted of the copolymerization of 2-hydroxyethyl methacrylate, with 3-(trimethoxysilyl)propyl methacrylate (organic phase), using benzoyl peroxide as thermal initiator and tetrahydrofuran as solvente. In the second step, the inorganic phase was obtained by mixing tetraethyl orthosilicate, etanol and water acidified with HNO3 (pH 1). The third stage consisted of mixing the inorganic phase with the organic one to obtain the hybrid. The samples were analyzed by Fourier Transform Infrared Spectroscopy, Scanning Eléctron Microscopy, Energy Dispersive Spectroscopy, Electrochemical Impedance Spectroscopy, potentiodynamic polarization curves and bioactivity assay. The results confirmed the formation of the hybrid and the achievement of a uniform and homogeneous surface coating on the alloy Furthermore, the corrosion resistance and bioactivity have been significantly improved. These results indicated that the Siloxane-p(HEMA) hybrid coating applied on Ti-6Al-4V alloy can act as a promising alternative for biomedical applications.

Acknowledgments:

CAPES, UFS, PPGQ, LPMH, NUPEG, NEREES, CMNANO.

References:

[1] S. V. Harb et al., “PMMA-silica nanocomposite coating: Effective corrosion protection and biocompatibility for a Ti6Al4V alloy,” Mater. Sci. Eng. C, vol. 110, no. December 2019, 2020, doi: 10.1016/j.msec.2020.110713.

Strontium ions enriched biomimetic coating on alumina/zirconia scaffolds obtained by gelcasting

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Porous bioceramics, which are known as scaffolds, can be used for bone tissue replacement or repair. In particular, alumina/zirconia (Al_2O_3/ZrO_2) bioceramics have excellent mechanical properties and chemical stability when compared to other materials. Due to their bioinertness, these biomaterials can be combined with calcium phosphates (Ca-P) to improve their bioactivity. Furthermore, the stimulation of bone formation through osteogenesis may be enhanced by incorporating strontium ions (Sr^{2+}) into the Ca-P structure. Herein, we studied the fabrication and characterization of Al_2O_3/ZrO_2 scaffolds (5 vol% in nano-sized ZrO_2 inclusions) using the gelcasting method and biomimetic coating with enriched with Sr^{2+}. Following scaffold fabrication, chemical surface treatment with phosphoric acid and coating with Synthetic Body Fluid (SBF) 5x more concentrated enriched with Sr^{2+} ions were performed for 14 days. We then characterized the scaffolds in terms of their morphology, porosity, and phosphate layer formed after incubation in SBF. In general, we observed the porous morphology of the scaffolds with high interconnectivity between the mostly spherical pores. Microtomographic analysis (µCT) revealed that approximately 60% of the pores present in the scaffolds had diameters between 120 and 350 µm, suggesting that the obtained scaffolds may promote bone tissue growth. Biologically interesting Sr-containing phosphates and Ca-P phases were observed on the surface of the scaffolds, suggesting that these materials are suitable for bone tissue engineering and bone repair and regeneration therapies.

Acknowledgements:

The authors gratefully acknowledge the financial support of grant 22/05031-0, São Paulo Research Foundation (FAPESP) and CAPES (Coordination for the Improvement of Higher Education Personnel) - Finance Code 001.

Study of the influence of cerium addition on the bioactivity of the CaO-Na_2O-P_2O_5-B_2O_3 vitreous system

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Bioactive borate glasses exhibit fast and complete conversion to hydroxyapatite (HA) when compared to bioactive silicate glasses [1-2]. Cerium-doped bioactive glasses can improve the performance of HA formation and fast bioactive response [3]. The aim of this study is to evaluate the influence of cerium oxide on the chemical durability of borate glasses in the vitreous system ($64B_2O_3$-$2P_2O_5$-(18-x)CaO-$16Na_2O$, with x = 0.5, 1, 1.5, and 2 mol% of CeO_2). The samples were synthesized by melt-quenching method and were analyzed by XRD and FTIR, before and after immersion in Simulated Body Fluid (SBF) for 28 days. The pH measurements were performed during the immersion period. Cytotoxicity was assessed on RAW-247 macrophages. The XRD diffractograms confirmed the amorphous nature of the samples and the FTIR spectra showed absorption bands characteristic of borate glasses. After imersion in SBF, the XRD diffractograms showed the presence of characteristic peaks of HA and $CePO_4$, with attention to the sample with 2 mol% CeO_2. The FTIR spectra of immersed samples showed the presence of phosphate groups (HA e $CePO_4$). The pH data indicate higher ion exchange between Ce-doped samples and SBF. The samples did not showed toxic behavior. The results showed that CeO_2 doped glasses improve the HA formation and demonstrate high bioactive behavior. Finally, the samples exhibited potential for tissue and bone regeneration.

Acknowledgment:This study was partly financed by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001. We are thankful to the Brazilian agencies CNPq, CAPES, FINEP, FAPEMA.

References:
[1] HENCH, L. L. Science, v. 295, n. 5557, p. 1014-1017 (2002)
[2] BALASUBRAMANIAN, P. et al. Journal of the European Ceramic Society, v. 38, n. 3, p. 855-869 (2018)
[3] ZHOU, G. et al. Materials, v. 12, n. 24, p. 4041 (2019)

Study of the thermal decomposition of telluric acid and coacervate with telluric acid

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Telluric acid, molecular form $Te(OH)_6$, is a white monoclinic crystal that is very soluble in water, consisting of octahedral molecules stable in aqueous solution because it is a weak acid [1]. Its thermal decomposition was studied by Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TG). The four phase transitions, all endothermic. According to the literature [2] the mass losses refer to the phase transitions that lead to the compounds $(H_2TeO_4)_n$, $H_2Te_2O_7$, $TeO_3$and TeO_2, respectively. A gradual change in the profile of the Raman spectra is observed, indicating the occurrence of the phase transitions described above. The large broadening of the bands reflects the low crystallinity of the compounds formed, making it difficult to assign bands. However, the telluric acid sample heat-treated at 500 °C exhibits a Raman spectrum similar to that of tellurium oxide (IV). From this material, $Te(OH)_6$, its calcium polyphosphate coacervate was obtained, which were analyzed by Raman spectroscopy and showed that telluric acid was incorporated into the phosphate network of the coacervate. From the coacervate with telluric acid, its thermal treatments were carried out, which were later analyzed by Raman spectroscopy.

Acknowledgments:

CAPES, CNPq, and FAPEMIG supported this work.

References:

[1] J. C., BART, v. 8, n. 2, p. 313-327, (1975);
[2] R. C., ROPP, p. 105-197, (2013)

Surface Biofunctionalization by Laser Texture followed Diamond-Like-Carbon Coating

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The high reactivity of blood with any non-endothelial/natural surface is a major challenge in the development of long-term circulatory assistance devices. Dedicated to blood contact surfaces were developed by manipulating the surface energy of pure titanium using nanosecond laser texturing followed by Diamond-Like-Carbon coating [1]. The difference in coating thickness on the textured surface generated two conditions: (a) super-plasmophobic surface, capable of preventing contact interaction with blood, and (b) super-plasmaphilic, promoting the anchorage of blood constituents susceptible to endothelialization. A 20-week in vivo experimental study demonstrated preliminary results and trends of how this behavior will be in the practice of mechanical circulatory support. Four groups with three animals were considered for each type of implants, one Reference Material and three different Test Samples. The implant was positioned in the abdominal aorta of 300 g Wistar rats and the effects of the hemodynamic interaction were evaluated without anticoagulant. After twelve weeks, the implants were extracted and subjected to qualitative analysis by low-vacuum scanning electron microscopy and chemical analysis by X-ray Energy Dispersion. Surface regions that remained in intense blood flow showed more significant interaction responses and those that remained in contact with the aortic wall, encapsulation by the endothelial tissue. The ultra-fast laser texturing process will need to be improved to ensure shear strength and applicability in parts with unconventional geometry. The coating with a greater thickness of diamond-like carbon showed an inhibitory effect on the endothelialization process, even under a super-plasmaphilic effect. Acknowledgements: Research Support FAPESP, CAPES and Company CloroVale. References: [1] BOCK E., et all. Laser-Treated Surfaces for VADs: From Inert Titanium to Potential Biofunctional Materials. AAAS BME Frontiers. 2022: 22-30.

Swelling and degradation properties of chitosan scaffolds containing grape seed oil for skin tissueregeneration

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Regenerative medicine is a prominent area aiming the development of biomaterials for application in the repairment of injured tissues. The skin tissue performs important functions for the maintenance of the body and its injury can lead to complications in the patient's health. Chitosan is a promising biomaterial for tissue regeneration due to suitable characteristics such as biocompatibility and biodegradability, cost-effective production, and processing versatility. Also, it is possible to incorporate other substances, aiming specific biological properties for the treatment of different biological tissues. Grape seed oil can be an interesting alternative, which can optimize the tissue regeneration process due to its antioxidant, anti-inflammatory and antimicrobial characteristics. Swelling and degradation are very important characteristics because they affect tissue-biomaterial interface and subsequent cell adherence and attachment. In this work, chitosan scaffolds containing grape seed oil in different proportions were obtained by 3D printing. The properties of swelling and degradation of the developed materials were evaluated up to 21 days. Theresults indicate that the presence of the grape seed oil reduced water absorption anddegradation rate of the chitosan scaffolds. Thus, the incorporation of such oil can modulatethe degradation of chitosan based materials, aiming wound dressing applications.

Acknowledgments

The authors thank the financial support from PROPES/UFABC (Pró-Reitoria de Pesquisa da Universidade Federal do ABC), FAPESP (Fundação de Amparo à Pesquisa do Estado de SãoPaulo) and CAPES (cod 001). UFABC research facilities are also appreciated.

Synthesis and Characterization of 1,10-Phenanthroline and Silver(I) Complex

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The complexes prepared from 1,10-phenanthroline have demonstrated antitumor potential since this ligand could bind with DNA [1]. Silver, in turn, has known its biological activity for thousands of years [2]. In this study, a complex of silver(I) and 1,10-phenanthroline was synthesized, reported in the literature as bis(1,10-Phenanthroline-N,N’)-silver(i) nitrate [3]. The material was synthesized using the slow solvent evaporation technique. X-ray diffraction technique (XRD) and Rietveld refinement, demonstrate that the material crystallizes an orthorhombic structure with the Fddd-space group. Thermal properties were studied using thermogravimetry (TG), differential thermal analysis (DTA), and differential scanning calorimetry (DSC) techniques. The material has shown thermal stability up to 373 K. The vibrational modes of the complex were measured using the Fourier Transform Infrared Spectroscopy (FTIR) technique. The results demonstrate that the material is promising for applications such as antitumor.

Acknowledgments:
We thank the FAPEMA, CAPES, CNPQ for their support in this research.

References:

[1] Bencini, A. and V. Lippolis, 1,10-Phenanthroline: A versatile building block for the construction of ligands for various purposes. Coordination Chemistry Reviews, 2010. 254(17): p. 2096-2180.
[2] Medici, S., et al., Silver coordination compounds: A new horizon in medicine. Coordination Chemistry Reviews, 2016. 327-328: p. 349-359.
[3] Li, P.-G., et al., Crystal structure of bis(1,10-phenanthroline-N,N')silver(I) mononitrate, [Ag(C12H8N2)2][NO3]. 2006. 221(3): p. 391-392.

SYNTHESIS AND CHARACTERIZATION OF COMPLEX BIS((μ_2-HYDROXO)-2,2'-BIPYRIDYL)-NITRATE-COPPER(II))

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Metal complexes can easily interact with proteins and nucleic acids through the complexation of metal ions with the ligand through electrostatic reactions and the formation of hydrogen bonds [1]. Thus, they have been used for a long time to treat diseases caused by different types of microorganisms, making it interesting to carry out studies to synthesize this type of material. They are heavily investigated due to their dual antimicrobial and antitumor activity. Thus, this work aimed to synthesize the complex bis((μ_2-hydroxo)-2,2' -bipyridyl)-nitrate-copper(II)) [2] by the slow solvent evaporation method and perform the characterization of the structure by X-ray diffraction (XRD), vibration by Fourier transform infrared spectroscopy (FT-IR) and thermal by thermogravimetric analysis (TG), differential thermal analysis (DTA) and differential scanning calorimetry (DSC). XRD at room temperature showed that the material crystallizes in a triclinic system with space group P1(2). The FT-IR spectrum confirms the complexation of Cu(II) with bipyridine, showing bands at 1372 and 1637 cm^{-1} attributed to the stretching modes of the bipyridine nitrogenatoms with the copper atoms. The thermal analysis of TG -DTA and DSC showed that thecrystal has thermal stability up to 200°C.

Acknowledgments:
We thank the CAPES, CNPQ, FAPEMA, PPGCM-UFMA, LDRX, and collaborators.

References:

[1] UMA, Varadaraja ELANGO, Munusamy; NAIR, Balachandran U. Copper (II) terpyridine complexes: effect of substituent on DNA binding and nuclease activity. European Journal of Inorganic Chemistry, p.3484-3490, 2007.

[2] TADSANAPRASITTIPOL, Amornphat; KRAATZ, Heinz-Bernhard; ENRIGHT, Gary D. The chemistry of dinitrato-2,2′-bipyridinecopper (II): preparation and characterization ofbinuclear complexes having a Cu (μ-OH) 2Cu and Cu (μ-N3) 2Cu core. Inorganica chimicaacta, v. 278, n. 2, p. 143-149, 1998

Synthesis and characterization of multifunctional mesoporous silica nanoparticles containing gold nanoparticles and gadolinium as theranostic system

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Mesoporous silica nanoparticles are well-known materials used as drug delivery systems due to their biocompatibility, well-defined pore diameter, and high surface area [1]. As well as gold nanoparticles are primarily studied as nanoplatforms for theranostics cancer due to their optical properties which can be used for diagnosis and treatment [2]. Moreover, gadolinium remains the most common MRI contrast agent to enhance the brightness of the region of interest [3]. This study used gold nanoparticles and gadolinium oxide incorporated into the silica matrix to design a theranostic system. The techniques used to characterize the nanocomposites were infrared spectroscopy (FTIR), ultraviolet-visible spectroscopy (UV-Vis), thermogravimetric analysis (TGA), nitrogen adsorption, scanning electron microscopy (SEM), dynamic light scattering (DLS), X-ray fluorescence (XRF), X-ray diffraction (XRD), and vibrating sample magnetometry (VSM). To confirm the presence of gadolinium and gold in the silica network, UV-Vis, nitrogen adsorption, XRF, XRD, SEM, and VSM techniques were used. The results showed that nanocomposites were successfully obtained and the incorporation of gold nanoparticles and gadolinium in the MCM-41 matrix was achieved, indicating the system has a potential application in cancer treatment and diagnosis.

References:
1. Meireles, I. B. da C. J. et al. Synthesis and characterization of gold nanorods coated by mesoporous silica MCM-41 as a platform for bioapplication in photohyperthermia. Nanotechnology 32, 505720 (2021).
2. Guo, T. et al. Gadolinium oxysulfide-coated gold nanorods with improved stability and dual-modal magnetic resonance/photoacoustic imaging contrast enhancement for cancer theranostics. Nanoscale 9, 56-61 (2017).
3. Li, Z., Guo, J., Zhang, M., Li, G. Hao, L. Gadolinium-Coated Mesoporous Silica Nanoparticle for Magnetic Resonance Imaging. Front. Chem. 10, 1-10 (2022).

Synthesis and characterization of the SiO_2-Na_2O-CaO-P_2O_5 glass-ceramic doped with MgO by the sol-gel method

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Bioactive glass-ceramics used in bone regeneration due to their bioactivity, biocompatibility and ability to form a hydroxyapatite layer [1]. Furthermore, syntheses containing MgO as a dopant demonstrated better adhesions, cell proliferation and cell differentiation [2]. The aim of this work is to synthe and characterize SiO_2-Na_2O-CaO-P_2O_5 glass-ceramic (SNCP) doped in different concentrations of MgO (0.5, 1 and 1.5 %mol). Samples of composition $50SiO_2$-$25Na_2O$-21CaO-$4P_2O_5$ (% mol) were prepared by the sol-gel method [3], with the reagents: $Na_2SiO_3.5H_2O$, P_2O_5 and $Ca(NO_3)_2.4H_2O$. The gel obtained was aged for 5 days at room temperature in a desiccator and dried in an oven at 60 °C/72 h. The powder was homogenized in a mortar with the aid of a pestle, both of agate, and were transferred to a Pt:5%Au crucible for heat treatment in a resistive furnace under the conditions: 200 °C/40h, 600 °C/5 h, 800 °C/3 h and 1000 °C/2 h. The heating rate was 10 °C/min, with slow cooling to room temperature. X-ray diffraction (XRD), scanning electron microscopy (SEM)and Fourier transform infrared spectroscopy (FTIR) were performed. XRD analysis showedthe formation of $Na_4Ca_4Si_6O_{18}$, $Na_6Ca_3Si_6O_{18}$ and Ca_2SiO_4 phases in the structure of pure and MgO-doped glass-ceramics. SEM analysis showed that the samples have particles of irregular shapes and varying sizes. The FTIR data showed bands at: 1082 and 1035 cm^{-1} ofSi-O-Si asymmetric axial strain and at 621 and 574 cm^{-1} of P-O angular strain. Preliminary results show the viability of the synthesis the SNCP glass-ceramic by sol-gel method withdifferent concentrations of MgO and a great potential for bone regeneration.

Acknowledgements:

The authors are thankful to CAPES and FINEP (ref. 0533/16)

References:

[1] ALADEL, B. A. et al. Int J Mech Eng. 10, 97-106 (2019)
[2] DRUZIAN, D. M. et al. Disciplinarum Scientia. 21, 169-178 (2020)
[3] ADAMS, L. A. et al. New Journal of Glass and Ceramics. 3, 11-15 (2013)

Synthesis, characterization, and anticancer activity of a coordination complex of cobalt(II) with 1,10-phenanthroline and malic acid ligands

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The study of coordination complexes of transition metals has been increasingly performed due to their promising biological activities. Cobalt stands out among metals due to its presence in vitamin B12 and its important role in the biochemical reactions of metalloenzymes [1]. Additionally, 1,10-phenanthroline is a formidable planar bidentate ligand that easily coordinates to transition metals, forming coordination complexes in a crystal lattice [2]. The aim of this work was to synthesize a coordination complex containing the cobalt(II) transition metal ion coordinated to the 1,10-phenanthroline and malic acid ligands (CoPhenMale), characterize, and study its thermal, vibrational, and biological properties, by means of the techniques of X-ray diffraction (XRD), differential thermal and thermogravimetric analysis (DTA/TG), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy, and anticancer activity tests. XRD results showed that the synthesizedcomplex crystalizes in the triclinic crystal system of the P1 space group. Rietveld refinementproved the crystalline structure since the parameters obtained are in a good agreement withdata available in the literature [3]. The DTA/TG results showed that the crystal presents agood thermal stability, with endothermic dehydration events. The analyses of the spectra provided a detailed attribution of the main bands related to the vibrational modes of thecomplex. Partial results of antitumor activity tests demonstrate a reduction of 60% of cellviability in breast cancer cell lines MDA-MB-231 and MCF-7 after 48 h of treatment. Fromthis study, we determined several properties of the material obtained and confirmed that itpresents promising possible applications as an antitumor agent.

[1] A. I. Stoica, M. Peltea, G. E. Baiulescu, M. Ionica, Jour. Pharma. Biomed. Analy., v. 36, p. 653, 2004.
[2] A. Bencini, V. Lippolis, Coord. Chem. Rev., v. 254, p. 2096, 2010.
[3] B. H. Toby, Powd. Diffrac., v. 21, p. 67, 2006.

Synthesis, Characterization and Biological Evaluation for Ruthenium (II) and Ruthenium (III) Coordination Compounds using N-(3-Pyridyl)-Isonicotinamide Ligand

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The present work shows the route of synthesis and the characterization of the complexes $[Ru(Cl)_3(H_2O)_2(3\text{-}pina)].5H_2O$ C1, $[Ru(Cl)_2(H_2O)_3(3\text{-}pina)]Cl.1/2H_2O$ C2 and $[RuCl(NH_3)(H_2O)_2(3\text{-}pina)](BF_4).1/2H_2O$ C3, with the linker N-(3-pyridyl)-isonicotinamide (3-pine), and its application in tests of microbicidal and phagocytic activities for Staphylococcus aureus (Gram positive) and Escherichia coli (Gram negative) bacteria. Characterizations by TG-DTG and TG-DSC show loss of C1 mass in three steps, related to the oxidative degradation process of (3-pin) with the formation of RuO_2 residue and error 0.337% (Exp.= 64.431%; Calc = 64.094%). C2 suffers mass loss in five stages with the formation of RuO_2 residue and error of 0.584% (Exp. = 71.080%; Calc. = 71.660%). C3, undergoes mass loss in six steps with an error of 0.926% (TG/exp= 74.451%; Calc= 73.525%) and RuO_2 residue [1]. Biological applications were carried out in C2 and C3, as for compound C1 it is insoluble in water. The viability analysis was performed for compounds C2 and C3 and free ligand and showed no toxicity against the PBMC cell. Microbicidal and phagocytic activity tests for bacteria (Gram positive) and (Gram negative)[2] indicate the antimicrobial and phagocytic potential of the compounds. Agreement: Federal University of Mato Grosso (UFMT); Materials Research Lab (MRL);Chronoimmunomodulation Laboratory and CAPES Palavras-chave: Rutênio, ligante 3-pina, atividade biológica. References. [1] Tarso, Moraes Fabricio, et al. Advances in Biological Chemistry 10., 86-98 (2020). [2] Galvão, Anderson Dourado, et al. Open Journal of Inorganic Chemistry 9., 11-22 (2019).

Synthesis, characterization, and vibrational study of binuclear maleatocopper(II) complex with phenanthroline

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Crystalline complexes containing transition metals and bidentate chelating compounds play primary roles in numerous biochemical processes [1]. Besides enhancing the biological activity of different ligands, copper complexes provide the development of antacid, anti-inflammatory, antiviral, antitumor, antimalarial, alzheimer''s treatment properties due to their neuroprotective action [2]. Thus, this work aimed to synthe and characterize binuclear copper(II) complexes with the ligands 1,10-phenanthroline and maleic acid to study their structural, vibrational, and thermal properties. The crystal was synthed with an equimolar ratio of the compounds by the slow solvent evaporation technique at room temperature. The crystalline complex containing two Cu2+ ions, two molecules of 1,10-phenanthroline, two maleates, and four water molecules (Cu2Fen2Male2) was characterized by X ray diffraction (XRD), differential thermal analysis, and thermogravimetric analysis (DTA/TG), Fourie transform infrared spectroscopy (FTIR) and Raman. The XRD results and Rietveld refinement confirms that the complex crystallizes in a monoclinic structure (space group C2/c), with four molecules per unit cell (Z=4). The lattice parameters showed good agreement with the literature [3]. The vibrational absorption and scattering modes exhibit the primary bands associated with the deformations and stretching of the Cu2+ ions with oxygen and nitrogen atoms to which they are bonded. The thermal analyses show that the complex has thermal stability up to 320K.

Acknowledgments: We thank the FAPEMA, CAPES, CNPQ for supporting this research.

References:

[1] Kumar, M., Parsekar, S. U., Duraipandy, N. et al., Inorg. Chim. Acta, v. 484, p.219, 2018.
[2] Iakovidis, I., Delimaris, I., Piperakis, S. M. Molecular biology international, v. 2011, p. 13, 2011.
[3] Li, Z. Y., Xu, D. J. Chiang, M. Jour. of Chem. Cryst., v. 35, p.615, 2005.

SYNTHESIS OF BARIUM HEXAFERRITE DOPED HYDROXYAPATITE SCAFFOLDS PREPARED BY THE FREEZE-CASTING METHOD

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Fractures and bone defects continue to be a problem that significantly affects the economy of the world and the patient's quality of life. The search for synthetic materials that are biocompatible, osteoconductive and osteoinductive has been a very promising alternative for the treatment of fractures and bone diseases [1]. Hydroxyapatite has characteristics similar to that of bone and its use has been disseminated in several studies, since the development of hydroxyapatite scaffolds for bone regeneration has been carried out. there.The objective of this article is to study the influence, effect and viability of thehydroxyapatite monolith through the freeze-casting technique and dop it with barium hexaferrite that will be produced by the sol-gel combustion method. The structure of hydroxyapatite doped with barium hexaferrite was investigated by thermogravimetric analysis (TGA), X-ray diffraction (XRD), compressive strength assay, infrared spectroscopy,gas adsorption method, scanning electron microscopy (SEM), microtomography, and Archimedes principle. By increasing the calcination temperature of barium hexaferrite, theoxidation process occurs, but when doping occurs, this phenomenon is not observed. Theporosity increased as the amount of barium hexaferrite increased, resulting in a more porous region at the top of the monolith and denser in the region that is in contact with thecopper dowel.

Acknowledgements:The Fundação de Apoio à Pesquisa do Distrito Federal (FAPDF) and the University of Brasilia (UnB) for the incentive to research.

References:

[1] M. R. Mohd Rosland et al., “The state of starch/hydroxyapatite composite scaffold in bone tissue engineering with consideration for dielectric measurement as an alternative characterization technique,” Materials, vol. 14, pages 1-17 (2021)

Synthesis of gadolinium-doped hydroxyapatite nanoparticles using a microwave-assisted hydrothermal method

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Bioceramics such as hydroxyapatite (HA) have been widely researched for biological applications due to their properties related to their chemical similarity with the inorganic phase of bones and teeth [1]. Furthermore, the flexibility of its structure allows a series of ionic substitutions, which gives it new properties. Doping with lanthanide ions has been widely investigated due to the similarity of the ionic radius of calcium (Ca^{+2}). In particular, gadolinium ions (Gd^{3+}) have been used in clinical practice as a contrast agent, in the form of metalorganic complexes, for nuclear magnetic resonance (NMRI). However, the Gd^{3+}ion has high toxicity when free in the bloodstream and, in the long term, individuals exposed to gadolinium-based contrast agents may have health problems related to the accumulation of Gd^{3+}in tissues and organs [2]. In this sense, due to the high stability of the HA structure, the production of HA nanoparticles doped with Gd^{3+}(n-HAGd) can be an alternative to toxicity problems. Among the different existing methods for HA synthesis (hydrothermal, precipitation, sol-gel, pyrolysis, hydrolysis, sonochemical, solid-state synthesis), microwave-assisted hydrothermal synthesis has the advantage of shorter synthesis time and more efficient heating. Based on the above, the objective of this work is the synthesis and characterization of HA nanoparticles doped with different concentrations of Gd^{3+}(n-HAGd). n-HAGd nanoparticles were synthed using the microwave-assisted hydrothermal method. The synthed nanostructures were characterized by XRD, TEM, FTIR, TGA, and VSM techniques. The results indicated the obtainment of carbonated n-HAGd with paramagnetic properties with the potential to be used as a contrast agent in NMRI.

References:
[1] Nasiri-tabrizi B., et al. Ceramics International, vol.49, p. 7142-7179 (2023).
[2] Akbas E., et al. Journal of Applied Toxicology, p. 1-15 (2022).

Synthesis of titanium-doped phosphate glasses and optical, mechanical and biological characterization for application in biomaterials.

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Phosphate-based bioglasses have been studied in recent years as a promising material for applications in dentistry and bone repair. These materials must have antibacterial characteristics and have the ability to induce bone formation. In this work, phosphate glasses with the structure $55P_2O_5-5ZnO-(30-x)Na_2O-10Al_2O_3-xTiO_2$(x=0;0.1;0.5;1 and 2mol%) were synthed using the melt tempering technique. The reagents, in powder form, were mixed, placed in a crucible and placed in an oven at a temperature of 1200°C. The molten material was poured into preheated molds at a temperature of 435°C and subsequently taken to the oven for annealing. Characterizations of MIR (Mid-Infrared), NIR (Near-Infrared), UV-Vis Spectroscopy were carried out in order to obtain the absorption coefficient and Band Gap energy information of the materials. Vickers microhardness measurements showed the difference in hardness between the synthed materials. Scanning Electron Microscopy (SEM) were carried out in order to observe the formation of apatite in the material. Finally, they were conducted to verify the cytotoxicity of the synthed glasses. The materials showed promise for application as bioglasses.

Acknowledgements: CNPq, CAPES, FAPEMIG, UFJF.

[1]NEEL, Ensanya A. Abou; *et al.* Mineralized nodule formation in primary osteoblasts culture in titanium doped phosphate glass and in-house prepared freeze dried demineralized bone extracts. Materials Chemistry And Physics, [S.L.], v. 276, p. 125425, jan. 2022.[2] NEEL, Ensanya A. Abou; *et al.* Remineralization potential and biocompatibility of titanium dioxide-doped phosphate glasses. Materials Letters, [S.L.], v. 309, p. 131456, fev. 2022.[3]NEEL, Ensanya A. Abou; *et al.* Antibacterial effect of titanium dioxide-doped phosphate glass microspheres filled total-etch dental adhesive on S. mutans biofilm. International Journal Of Adhesion And Adhesives, [S.L.], v. 108, p. 102886, jul. 2021.

SYNTHESIS, STRUCTURAL, THERMAL, VIBRATIONAL PROPERTIES AND DFT STUDY OF COPPER(II), 1,10-PHENANTHROLINE AND ISOLEUCINE COMPLEX FOR POSSIBLE APPLICATION AS ANTITUMOR

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The present work seeks to explore the properties of metal complexes in order to increase the efficiency of DNA strand cleavage by means of the binding affinity of the metal complex with DNA. 1,10-phenanthroline (fen) is able to bind to DNA and L-isoleucine is essential for the development of skeletal muscle tissue [1]. Based on this possibility, this work aimed to synthesize by the slow solvent evaporation method and characterize the physical and chemical properties of copper(II) crystal, 1,10-phenanthroline and Isoleucine. The obtained crystalline materials were characterized by X-ray diffraction (XRD), thermogravimetric analysis (TG), differential thermal analysis (DTA), FT-IR spectroscopy, Raman and DFT study. The crystal was obtained and its structure confirmed by refinement using the Rietveld method. The data are in agreement with that reported in the literature [2]. The TGand DTA analyses showed that the material has thermal stability up to 323 K, higher thanthe human body temperature, undergoing phase transformation by dehydration due to theexit of free water from the compound. The FT-IR and Raman analysis evidenced the complexation of the copper(II) ion with the 1,10-phenanthroline and Isoleucine molecules, since the vibrations observed are associated with these molecules. The chemical reactivitydescriptors obtained by the DFT method indicate that the complex has the potential toparticipate in chemical reactions and interact with biomolecular targets. These results suggest that the complex has the potential to be used in chemotherapeutic treatment ofcancer cells.

Acknowledgements:
We thank the FAPEMA, CAPES, CNPQ, PPGCM-UFMA, LDRX, and collaborators.

References:

[1] NELSON, David L.; LEHNINGER, Albert L.; COX, Michael M. Lehninger principles of biochemistry. Macmillan, 2008. [2] Chen Shi, Le Xueyi, Zhou Xiaohua, Liu Xiaoping, Liu Guang CCDC 213583: Experimental Crystal Structure Determination, 2005, DOI: 10.5517/cc757sd.

Technical and economic viability of 420 steel forcutting tubes in keratoprosthesis surgery.

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The use of metallic materials in ophthalmology is constant, both in the surgical area and in the clinic, present in the treatment of ocular trauma, infectious diseases or in the simple correction of refractive errors, with this the search for improvements and improvement of the tools becomes increasingly necessary . In ophthalmology, a stainless steel trephine is used, martensitic stainless steel of the AISI-420 type is widely used in the manufacture of surgical tools, it is designed for performing corneal surgeries, it needs to have a good cutting edge, a thin and symmetrical blade causing a perfect flattening in conformity with the córnea. Because it has a high demand and high costs, it presents a problem for the health system, whether public or private. This work was motivated by the high cost of the tool and because it has a short useful life in the surgical procedure, it will evaluate the chemical composition, the hardness, the microstructure, the surface treatments and the wear resistance, and its objective is the improvement of stainless steel 420 via plasma nitrogen deposition under different conditions forming a new layer on the surface of the material, aiming at reducing the cost and improving the useful life of this cutting tool, withthis characterizations for evaluation will be carried out to qualify the best possible technique accordingly with results of greater hardness, less wear and improved product life,obtaining the best performance and carrying out its functions correctly.

Acknowledgements:
To Professor Dr. Rômulo Ribeiro Magalhães de Sousa, coordinator of LabPlasma, located at Universidade Feral do Piauí, for his contribution in the deposition process and to everyonewho added to the work.

References:
[1] Rodrigues, C.A.D., Enokibara, F., Leiva, T.P., Nunes, I.A., Rollo, J.M.D.D.A. (2009). Quality of AISI-420 type martensitic stainless steel used in the manufacture of surgical tools. Rem: Revista Escola de Minas, 62, 475-480.

Temperature dependence to exchange native ligands in post-synthesis reactions for zirconia nanocrystals

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From a chemical synthesis route, the surface ligands that cover nanocrystals (NCs) are required to produce nanomaterials with controlled growth and stabilized surface. Changing the surface ligands in a second synthesis step has been used to modify the surface of nanomaterials and achieve desired behaviors without changing the characteristics of the produced NCs[1]. In this work, ZrO_2 NC was synthesized using benzyl alcohol as solvent and zirconium butoxide in butanol as a metallic precursor. NCs with size around 5 nm, narrow size distribution and cubic crystalline phase were obtained. Native capping ligands from the synthesis were identified as benzoates and butanoates from the solvents used in the synthesis [2]. Ligand exchange was performed by immersion in excess synthesis solvents at room temperatures and near the boiling point of the solvents. Characterization by FTIR spectroscopy indicated that temperature plays a fundamental role in removing native ligands from the synthesis, and it was also possible to identify different modes of binding benzoate and butanoate ligands with NCs surface. Thermogravimetric analyses allowed quantification of the ligands and confirmed different binding forces with the surface of the NCs. We conclude that for ligands precursors with closed pKa value, high temperatures are required to exchange almost all native ligands from NCs surface without changing the crystalline phase. Those results contribute to the study of the composition of the binders in the ZrO_2 NCs and reveal that the surface modification treatment used can be adjusted to control the covering capping agents in NCs.

Acknowledgements:

CAPES, FAPES, CNPq e FINEP-ANP PRH 53.1 and Lab NCQP.

References:

[1] R. Deshmukh and M. Niederberger, Chem. - A Eur. J., vol. 23, no. 36, pp. 8542-8570, 2017

[2] M. S. Santos, C. J. Dalmaschio, and J. C. C. Freitas, CrystEngComm, vol. 22, no. 10, pp. 1802-1811, 2020

THERMAL DEGRADATION STUDY OF GREENISH BROWN PROPOLIS MICROCAPSULES.

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Propolis is a resinous substance of complex composition, formed by gummy and balsamic material, collected by bees from different types of trees, this product has been the subject of pharmacological studies due to its antibacterial, antifungal, antiviral, anti-inflammatory, antioxidant, and antitumoral properties[1]. Microencapsulation prevents the loss of substances by oxidation or volatilization, as well as controlling the release of aromas, and loss of nutrients, and preserving the color and flavor of the encapsulated agent[2]. In this work, the study of thermal degradation in greenish-brown propolis microcapsules obtained using a Spray Dryer was carried out, the objective was to evaluate the stability of the microcapsules about the protection of the active compounds present in the extract, such as the content of flavonoids and phenols. The extract and the microcapsules containing greenish-brown propolis extract presented in their composition full flavonoid content of 5.15% and 1.13%, total phenols of 14.16 and 4.81% in terms of gallic acid, total phenols of 16.53% and 6.00% in terms of catechin, antioxidant activity with IC50 values of 13.77 μg/mL and 60.80 μg/mL, respectively, and thermal analysis showed the thermostability of the studied extracts. According to the TG analyses, the mass loss was 19.152% from 26.02 to 215.90 ºC, in the first moment, and afterwards, the mass loss was 52.486% from 215.90 to 531.53 ºC. These data mean, respectively, the loss of volatile compounds and the decomposition of the encapsulating material. It is worth mentioning that the formation of greenish-brown propolis microspheres has not yet been reported in the literature, demonstrating this work's originality. ACKNOWLEDGEMENTS: CNPQ, FAPEAL, and CAPES.[1]MARCUCCI, M.C.Propolis: chemical composition, biological properties, and therapeutic activity.Apidologie,v.26,n.2,p.83-99,1995. [2]PEZZIN,A.P.T.;MEIER,M.Microencapsulation: innovation in different areas.n.july,2015.

THE ROLE OF BaO ON BIOACTIVITY AND RADIOPACITY OF BOROPHOSPHATE GLASS FOR BONE REGENERATION AND SHIELDING IONIZINGRADIATION

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The present study reports the preparation of borophosphate glasses (40-x/2)B_2O_3-(25-x/2)P_2O_5-7CaF_2-20Na_2O-8CaO-xBaO (x=0, 5, 7.5 and 10 % in mol) in order to investigate the role of BaO on its physical, radiopacity, and bioactive properties. The samples were synthesized by melting-quenching technique and submitted to bioactivity test in SBF for 28 days. Before SBF immersion, the samples were characterized by XRD, FTIR, radiography; ionizing radiation shielding parameter were also calculated. After SBF immersion, pH measurements, XRD and FTIR were also performed to investigate the bioactivity of the glasses. Cytotoxicity test were user to evaluate the influence of BaO in cell viability. Results before SBF immersion, XRD proves the amorphous nature of the samples, FTIR indicated structural changes in the glasses with the increase of BaO. It was possible to compare the radiopacity of the 0 mol% BaO sample with aluminum and dentin of the same thickness (1mmEq/Al). The other samples have the value of 1.68; 2.29 and 2.90 mmEq/Al (5, 7.5 and 10 mol% with BaO, respectively)[1]. The mass attenuation coefficient (MAC) shows that the attenuation of the radiation in the low energy region of X-rays (60KeV) is notably increased with the BaO content and reaches a value of up to 16 times that of common concrete for the sample with 10% BaO, Results after SBF immersion, final measured pH value between the samples decreased with the increase in the concentration of the BaO, indicating a decrease in the ionic release rate with the increase in this concentration. The XRD and FTIR analyses, shows the presence of hydroxyapatite and fluorapatite phases. The cytotoxicity results indicate that all prepared samples are not cytotoxic up to 10 mol% of the BaO.

Acknowledgments:CNPq and CAPES for financial support of this study.

References:

[1] R.B. Ermis, D. Yildirim, G. Yildiz, O. Gormez. Eur J Dent. 8 (2014) 342-347. https://doi.org/10.4103/1305-7456.137644.

Titanium coating with calcium titanate by sol-gelmethod: the influences of calcium amount and temperature

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Bioceramics have been considered as a component of devices for bone fractures and defects correction, as they provide good adsorption and cell growth. Because of these properties, they are used as surface coatings on metallic implants. Calcium titanate stands out because it promotes apatite precipitation and optimizes bone fixation time to the titanium [1]. Also, it can be applied in association with hydroxyapatite. The objective of this work is to prepare calcium titanate films on titanium and studying the influences of calcium amount and temperature on the crystalline structure, crystallinity of films and surface charge. Samples of commercially pure titanium (cp-Ti., ASTM F67 GR2) were polished and treated with diluted hydrochloric acid. Sols with Ca:Ti molar ratio of 1:2, 3:4, 1:1. The titanium plates were coated by dipping into the sols, hydrolyzing under atmospheric humidity and heat treating in the temperatures between 400ºC and 600ºC. The samples were submitted to two characterization techniques: X-Ray Diffraction (XRD) and Surface Zeta Potential measurements. XRD analysis showed that the presence of crystalline calcium titanate (PDF: 42-423) was more evident when the films were prepared in a 3:4 ratio and calcined at 600°C, indicating that the preparation method used is effective to coat the titanium with calcium titanate. The study of surface zeta potentials indicated that both the variation in molar concentrations and temperature influenced the surface charges This study allowed to identify the characteristics of the surfaces as a function of synthesis conditions, which will be useful to choose the adequate methods to deposit on the modified substrate.

References:

[1]Abdulkareem and Anjeel; Effect of annealing temperatures on the structural and crystalline properties of CaTiO3 powder synthed via conventional solid-state method. Materials Today: Proceedings, 3rd International Conference on Materials Engineering Science. v. 42, p. 2674-2679, (2021).

Zinc scaffold synthesis by powder metallurgy for application as biomaterial

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The ideal bone support material should be osteoconductive, have the ability to form blood vessels over and around the implants within a few weeks of implantation to support the transport of oxygen, nutrients and waste products. biomaterial. Along with the mechanical properties corresponding to bone, the scaffold must be able to degrade over time in vivo ata controlled rate of resorption, creating the space for new bone tissue to Grow[1], these materials are called bioresorbable [2]. In this context, zinc alloys manufactured by powder metallurgy were synthesized and porosity parameters by gas adsorption method, microtomography and Archimedes principle, hardness by Virkers hardness measurements, microstructure by X-ray diffraction (XRD) and composition by X-ray fluorescence.

Acknowledgments

The Federal District Research Support Foundation (FAPDF), the University of Brasilia (UnB) and the Federal Institute of Pará (IFPA) for encouraging research.

References

[1] Parai, R., Bandyopadhyay-Ghosh, S. Engineered bio-nanocomposite magnesium scaffold for bone tissue regeneration. Journal of the mechanical behavior of biomedical materials, v. 96, p. 45-52, 2019.

[2] Hernández-Escobar, D., Champagne, S., Yilmazer, H., Dikici, B., Boehlert, C. J., Hermawan, H. Current status and perspectives of zinc-based absorbable alloys for biomedical applications. Acta biomaterialia, v. 97, p. 1-22, 2019

F-Nanomaterials in Medicine, Nanotoxicology and Nanoregulation: Exploiting the Nano-Bio Interface for Clinical Translation

(Cancer) Theranostics with (Intrinsically) Radiolabeled Nanomaterials

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Radiolabeled nanomaterials have gained tremendous interest over the last 2 decades, which can play diverse roles in imaging, image-guided drug delivery, as well as theranostics of a number of diseases such as cancer. Some recent examples of radiolabeled materials from our recent work will be briefly described in this talk.

Although chelator-based radiolabeling techniques (commonly used for labeling nanomaterials with radiometals such as ^{64}Cu/^{89}Zr) have been used for decades, concerns about the complexity of coordination chemistry, possible alteration of nanomaterial pharmacokinetics, and potential detachment of radioisotopes have driven the need for developing a simpler yet better technique for future radiolabeling, which may facilitate future clinical translation.

The emerging area of intrinsically radiolabeled nanomaterials can take advantage of the unique physical and chemical properties of well-selected inorganic or organic nanomaterials for radiolabeling, and more importantly, offer an easier, faster, and more specific radiolabeling possibility to facilitate future clinical translation. Generally speaking, the four major categories of intrinsically radiolabeled nanomaterials include: 1) hot-plus-cold precursors, 2) specific trapping, 3) cation exchange, and 4) proton beam activation.

Representative examples of these strategies will be briefly illustrated in this talk, with the main focus on our own work that involves the radiolabeling of a variety of nanomaterials via “specific trapping”. The nanomaterials investigated in our laboratory include silica/carbon-based nanomaterials, COVID-19 nano-vaccines, multifunctional/multimodal hybrid nanomaterials, iron oxide nanoparticles, micelles, DNA nanostructures, etc.

Invited Speaker: Shedding Light on Ag2S Nanoparticles: Imaging, Sensing, and Beyond

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The use of luminescent nanoparticles for imaging and therapeutic purposes has gained significant attention in recent years. In this study, we present experimental data both on sensing capabilities as well as the pharmacokinetics of Ag2S nanocrystals of varying sizes, charges, and surfaces. These nanoparticles can be excited in the first near-infrared window and emit in the second, making them a promising candidate for biological applications. Our results show different patterns of distribution and a wide range of applicability for targeted imaging strategies. These findings could pave the way for the development of new imaging tools and therapies using Ag2S nanocrystals.

Nanoparticle-mediated immune suppression ofmetastatic cancer

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By virtue of size and coating, nanoparticles interact with innate immune cells. These interactions can stimulate immune signaling to initiate adaptive immune responses. Here, we explored such a potential shown by iron oxide nanoparticles coated with hydroxyl ethyl starch using transgenic and syngeneic metastatic breast cancer mouse models. Mouse models comprised Human HER2 overexpressing transgenic mice (HuHER2 in FVB/N background) that spontaneously develop primary breast tumors and lung metastasis (40-50%); and, orthotopic implanted Py230 cells growing in C57BL/6 mice that also spontaneously metastasize to lungs (30-40%). Tumor-bearing mice injected intravenously with Bionized Nano ferrite nanoparticles (BNF) at a non-toxic, human equivalent dose of 5mgFe/mouse into their tail vein displayed significant inhibition of tumor growth and complete inhibition of metastasis. In depth molecular analysis showed that the Toll Like Receptor (TLR) pathway mediated immune signaling was altered by BNF mostly in tumor and in nanoparticle associated cells collected from all analyzed tissues. BNF activated TIR-domain-containing adapter-inducing interferon-β (TRIF) culminating in phosphorylation and translocation of Interferon Response Factor 3 (p-IRF3) to the nucleus. This in turn activates interferon-β mediated antiviral response resulting in T cell stimulation. Requirement of TRIFin demonstrating anti-tumor immune effects was confirmed using TRIF knockout mice which displayed a complete reversal of primary tumor growth inhibition by treatment with BNF. These findings open a new direction to explore iron oxide nanoparticles as immune modulators with anticancer effect and their potential as TLR3 agonists for treating metastatic breast cancer.

Surface modification of nanoparticles: disguise, blendin or stand out?

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Several approaches to circumvent issues related to liposomes stability, circulation time or biointeractions are based on membrane surface modifications, being PEG the most widely used. However, PEG is also linked to a number of unwanted outcomes, such as accelerated blood clearance and other adverse reactions. We hypothesize that biomimetic liposomes, incorporating cell membrane derived components enriched with synthetic lipids might be a promising approach for specific nanomedicine applications. We have prepared biomimetic vesicles by combining specific cell membranes (erythrocytes or tumor cells) with synthetic or purified phospholipids using freeze-thaw cycles and extrusion protocols. Membrane properties and lipid dynamics were characterized by biophysics assays (EPR) and their performance was measured in vitro and in vivo (tumor murine model). Biomimetic hybrid liposomes present biophysical properties directly modulated by their synthetic lipid content. Biomimetic vesicles prepared with tumor cell membranes are able to induce immune response in mice following different cancer vaccination protocols leading to a reduced number of metastasis and increased animal survival rates. Biomimetic nanocarriers combining synthetic or purified phospholipids and different cell membranes into a hybrid nanovesicle can exhibit controllable properties derived from the biomembrane (such as self-markers and other proteins) and from added lipids, opening several possibilities for tuning specific features. These systems are excellent candidates as a nanomedicine platform since the insertion of purified or synthetic lipids in their preparation is one additional step towards scalability and reproducibility.

ANTIANGIOGENIC AND ANTITUMORAL ACTIVITY OF BIOMIMETIC DELIVERY SYSTEMS USING THE CHICKEN CHORIOALLANTOIC MEMBRANE (CAM) ASSAY

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Glioblastoma (GBM) is a prevalent malignant brain tumor with a high recurrence and mortality rate, with an overall median survival rate not longer than two years1. The limited prognosis of GBM is usually related to tumor heterogeneity, drug resistance, and insufficient brain drug delivery, as a result of the restricted access to the tumors imposed by the blood-brain barrier (BBB)2. In this regard, engineered biomimetic nanosystems have overcome important shortcomings over conventional therapy, providing significant features, especially in terms of therapeutic activity3. Here, we hypothesize that a biomimetic nanostructured delivery system based on polylactic/glycolic acid (PLGA) for temozolomide (TMZ) encapsulation should provide significant improvements in therapeutic outcomes regarding the antiangiogenic and antitumoral activity. Therefore, the U251 glioma cells (2x106 cells) were implanted into the CAM using maxgel support to perform a tumor mass. Tumor area and perimeter measurements were carried out before and after treatment and the CAMs with tumors were excised and imaged for blood vessel quantification. Our results indicate the antiangiogenic potential and tumor reduction due to biomimetic nanosystems treatment. This strategy may represent a potential treatment for GBM in future clinical applications improving therapeutic outcomes.

Acknowledgments: this study was supported by Sao Paulo Research Foundation (FAPESP, Brazil) Grant 2019/25645-0.

References: 1Sampson, J. H. et al. HHS Public Access. 20, 12-25 (2021).; 2Groothuis, D. R. The blood-brain and blood-tumor barriers: A review of strategies for increasing drug delivery. Neuro. Oncol. 2, 45-49 (2000).; 3Ferreira, N. N. et al. Modulating chitosan-PLGA nanoparticle properties to design a co-delivery platform for glioblastoma therapy intended for nose-to-brain route. Drug Deliv. Transl. Res. (2020).

Biomaterials based on mesoporous silica nanoparticles and hydroxyapatite for controlled DOX release

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Mesoporous silica nanoparticles (MSN) have been explored in biomedical field due to the possibility of trapping molecules within their ordered pore channels. Our research group have been already exploring this material using it as a nanoreservoir in the development of nanovalves for cancer treatment[1]. Because MSN structure is very stable, hydroxyapatite (HAP) can be incorporated into its structure aiming to improve MSN biodegradability and biocompatibility by enhancing MSN solubility in acid environment. Here, we propose two different biocompatible materials based on mesoporous silica and hydroxyapatite by using two different synthetic procedure [2,3]. Both biomaterials were compared with pure MSN. The first biomaterial, named MSN-HAP, displayed separated phases, in which MSN had a smaller size than pure MSN (50 versus 100 nm) and HAP presented an elongated shape. The second material, named HAP@MSN, showed a core-shell structure, where the MSN phase formed a shell around HAP phase. Also, the biomaterials presented similar BET specific areas and drug loading, but different pore arrays, without the hexagonal porous structure commonly observed for MSN. This fact was directly translated on DOX kinetic release behavior. The porous disorganization provided to the materials MSN-HAP andHAP@MSN a lower DOX release profile in comparison to pure MSN, at pH = 4.5, which is desirable for drug delivery systems. Moreover, as a result of the presence of HAP in a separated phase, MSN-HAP showed the lowest release because HAP particles could practically block the pores, hampering the drug release. In vitro studies against esophageal cancer cells TE-1 are being performed.

Acknowledgments:
UFF, CAPES, CNPq, FAPERJ, PPGQ-UFF, LAME, LAMATE

References:

[1] A. F. M. da Silva, et al. ACS Applied Nano Materials, 5, 10, 13805 - 13819 (2022)
[2] X. Hao. et al. ACS Nano, 9, 10, 9614-9625 (2015)
[3] R. K. Lenji, et al. Ceramics International, 43, 12857 - 12862 (2017)

Biomimetic magnetic nanocarriers for thermal nanomedicine

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Iron oxide-based nanoparticles have several clinical applications, such as anemia treatment, MRI contrast agents and magnetic hyperthermia. In this presentation we investigate the theranostic applications of membrane-coated magnetic nanoparticles. Distinct cell membranes are used for surface coating the nanoparticles namely erythrocyte, B16F10 (melanoma), 4T1 (breast cancer) and GL261 (glioblastome). Two magnetic nanoparticles are investigated, maghemite and Mn-ferrite. Nanoparticles were characterized by several techniques (TEM, XRD, NTA, DLS, VSM, among others). Distinct magnetic nanocarriers are prepared and their properties for thermal nanomedicine compared namely magnetic fluids, magnetoliposomes, magnetic hybrid liposomes, and biomimetic magnetic vesicles. T1 and T2 magnetic resonance imaging agent properties are obtained with a 1T MRI pre-clinical equipment. For some vesicle samples we also added IR-780 dye in the membrane for near-infrared imaging applications. Magnetic hyperthermia (MH) and photothermal therapy (PTT) data are reported at clinically relevant experimental conditions. We obtained the nanocarriers photothermal conversion efficiency parameter and demonstrate that the photostability depends on nanocarrier design. Preliminary results of MRI thermometry using the PRF method and the potential of IR-780 for luminescent thermometry during PTT are also reported. The data indicates that the biomimetic magnetic nanocarriers have great potential for cancer therapy.

Buffering effect of cell membrane-coated ZnOPEI nanoparticles on pancreatic cancer under hypoxia conditions

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Some cancers can lead to malignant progression and metastasis if diagnosed late. Advanced-stage tumors are highly vascularized but unbalanced, and in this case, the cells grow rapidly and undergo anaerobic respiration [1]. As a result, high concentrations of lactate are produced and the pH of the medium is lowered, known as tumor hypoxia condition [2]. Highly basic molecules/polymers (Polyethylenimine - PEI) can destabilize tumor metabolism, leading to tumor mass reduction and/or steady progression. In this study we focus on the synthesis of organic-inorganic positive hybrid nanoparticles vectorized using liposomes or real membrane, for action in the hypoxic tumor environment. The ZnO quantum dots was synthed by sol-gel methodology with their surface modified using GPTMS crosslinking with PEI polymer being developed a luminescent cluster nanoparticle with a compelling buffering characteristic. The buffering effects was confirmed by tritation carrying between 12 - 2 pH, showing two effects, first with protonation of NH2 groups of PEI and then with degradation of ZnO-GPTMS. Interestingly, ZnO degrades at low pH, being a possible hypoxia biomarker. The use of liposomes coated with biotin-modified F127 proved to be efficient in internalization and also showed greater stability than conventional liposomes. We also developed a nanoparticle covered with a real cell membrane (PANC-1 cell membrane), revealing a potential nanovector for application in personalized medicine. In this case, we performed for the first time an evaluation of the buffer effect on lactate inthe cells, ROS generation, and cell death. Our studies showed that cell viability decreased atlow concentrations and that the ROS production and intracellular lactate storage were dose-dependent and triggered cell apoptosis.

Acknowledgment: CAPES, CNPq and FAPESP (Grant number 17/22056-9)

[1] Faubert B, et al. Science. 2020; 368:6487.
[2] Cruz KG, et al J. Front Oncol. 2019; 1; 9.

Cell-Penetrating Peptide Nanotechnology: Opportunities in the Development of InnovativeTherapeutics

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Cell-penetrating peptides (CPPs) are bioactive molecules envisaged for the future development of nanotherapies. These peptide sequences, usually constituted by short strands of amino acids ranging from 5 to 30 residues, exhibit a strong ability to translocate through biomembranes, making them ideal candidates for the production of non-viral vectors in various applications, including gene therapy. In this communication, I will present recent studies carried out in our laboratory on the structure of DNA vectors based on CPPs that explored the design, characterization, and cell interaction of synthetic CPPs[1-3]. The structural features of different classes of CPPs will be highlighted, with special attention to hydrophobic CPPs that exhibit low cytotoxicity and high amyloidogenic propensity. Advanced methods of structural characterization of these systems will be discussed, with a focus on high-resolution small-angle scattering and AFM-based infrared spectroscopy. A special focus will be given to the need for interdisciplinary research at the interfaces of Physics, Chemistry, and Biology for the future development of peptide-based nanotherapy. Finally, the consequences of using these materials in the creation of new advanced materials will be highlighted, with examples pointing to future directions of development.

Acknowledgments: This research has been primarily supported by the Sao Paulo Research Foundation (FAPESP, #2022/03056-6 and #2019/19719-1) and the Brazilian National Council for Scientific Research and Development (CNPq, # 307443/2022-1).

References:

[1] *ACS Appl. Bio Mater.* **2021**, 4, 8, 6404 (https://doi.org/10.1021/acsabm.1c00601);
[2] *Soft Matter* **2020**, 16, 4746 (https://doi.org/10.1039/D0SM00347F);
[3] *J. Mater. Chem. B* **2020**, 8, 2495 (https://doi.org/10.1039/C9TB02219H).

CELLULOSE NANOCRYSTALS FOR ANTICANCER DRUG DELIVERY

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Compared to conventional therapeutics in cancer treatment, nanoparticle-based drug delivery has specific advantages, such as the ability to transport and release a high quantity of drug to a precise target, an improved stability and biocompatibility, enhanced permeability and retention effect, good pharmacokinetics and a role in overcoming cancer-related drug resistance. Among the several nano-systems under investigation for drug delivery, cellulose nanocrystals (CNCs) offer interesting opportunities as innovative nano-carriers in the oncological field for their versatility, easy tunable surface according to the nature of the loaded drugs. Moreover, they are suitable for clinical use thanks to their biocompatibility and biodegradation. Cellulose nanocrystals (CNCs) were made by an easy procedure wherein carboxylic groups are introduced into never dried cellulose pulp by oxidation at alkaline pH using 2,2,6,6-tetramethylpiperidine-1-oxyl radical (TEMPO) as a catalyst, followed by dialysis and autoclave sterilization. At the end of this procedure, a transparent suspension of rod-like CNCs is obtained. The obtained size is rather independent on the plant source and it is well known to fit in the range 3-5 nm of width and 200-500 nm of length. After the physical characterization by TEM (Transmission Electron Microscopy), DLS (Dynamic Light Scattering), and AFM (Atomic Force Microscopy) to investigate the morphology and the size, the carrier was loaded with anticancer drugs (e.g. doxorubicin). Drug release was studied in PBS (phosphate buffer saline) and water solution and resulted in an efficient release rate.Moreover, their cytotoxicity was evaluated in vitro on human cancer cell lines (prostate and lung cancer cells, i.e., PC3 and A549, respectively) and compared to the corresponding free drug.

Cubic phase nanoparticles in the presence of lysozymeand curcumin: A structural approach

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The study of drug delivery systems has gained much attention in recent years due to the advantages of using such systems, such as the possibility of controlling the amount of drug delivered to the recipient organism. Cubic lipidic nanoparticles, known as cubosomes, show promise within this perspective. This work aims to investigate physical characteristics as , concentration, and structure of cubosomes, encapsulating lysozyme and curcumin in different regions of this nanoparticle. Small Angle X-ray Scattering (SAXS) data were obtained at MAX IV synchrotron, while Dynamic light scattering (DLS) and Nanoparticle Tracking Analysis (NTA) data were obtained at LNLS/CNPEM. Also, a cell viability test was made, to obtain the drug''s activities against human breast cancer cells (MDA-MB-231) and human epidermal keratinocyte (HaCat). DLS and NTA results show that these cubosomes have an average of 200(2) nm, and an average concentration of 1.00(3)x1012 part/ml. SAXS curves show that the inner cubic structure of cubosomes is kept unaltered in the presence of increasing lysozyme and curcumin concentrations. Cell viability test show promising results for all the dosages in both cells, up to 70%. In short, our work intends to provide a study of structural and functional characterization about this system, and further, other analysis will be made to achieve them.

Acknowledgments:

CAPES, FAPESP, CNPQ, CNPEM, USP

References:

[1] GABALLA, Sherif A.; EL GARHY, Omar H.; ABDELKADER, Hamdy. Cubosomes: composition, preparation, and drug delivery applications. Journal of advanced biomedical and pharmaceutical sciences, v. 3, n. 1, p. 1-9, 2020.

[2] HINTON, Tracey M. et al. Bicontinuous cubic phase nanoparticle lipid chemistry affects toxicity in cultured cells. Toxicology Research, v. 3, n. 1, p. 11-22, 2014.

[3] UMAR, Hassaan et al. Cubosomes: Design, Development, and Tumor-Targeted Drug Delivery Applications. Polymers, v. 14, n. 15, p. 3118, 2022.

Design of luminescent SiO_2/EuTTA/ZIF-8 nanocomposite loaded with uvaol for use as drug delivery platform for treatment of pulmonary diseases

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Porous nanomaterials (PN) acting as drug delivery system have gained prominence in recent years Several types of PN acting as vehicles have been described, including compounds based in Zeolitic Imidazolate Framework-8 (ZIF-8) and its composites [1]. Urs-12-ene-3,28-diol (uvaol), has been one of the most interesting natural drugs due to its relevant inhibitory activity, effects on antigen-induced lung inflammation that are shown to be potentially useful for treating Chronic Obstructive Pulmonary Disease (COPD) [2]. In this work, a luminescente drug delivery nanocomposite based on the porous ZIF-8 combined with a Europium complex (EuTTA) to obtain the **EuTTA/SiO_2/ZIF-8**was used for uvaol release. XRD for **EuTTA/SiO_2/ZIF-8**showed characteristic planes of the ZIF-8 (I-43m space group) corresponding to the crystallographic data [3]. FTIR spectra revealed the signals: C=N (1520-1290 cm^{-1}), asymmetric (1601 cm^{-1}) and symmetric (1403 cm^{-1}) νCOO^-stretches from the imidazole ligand and the EuTTA complex, respectively. In addition, the emission spectrum revels the caracteristic f-f transitions$^5D_0 \rightarrow {}^7F_{0-4}$from Eu^{3+}ion, which are analogousto isolated EuTTA. Uvaol release assay was performed in PBS pH = 7.4 and revealedthat **EuTTA/SiO_2/ZIF-8**achieves rapid release (≈ 52%) after 8h. In addition, MTT assay using J774 macrophages showed viability greater than 85%. In short, the recent results obtained indicate that **EuTTA/SiO_2/ZIF-8**presents itself as a promising platform for use as avehicle for drug delivery of uvaol.

Acknowledgments: TecNano, CNPq, CAPES, FACEPE and FAPEAL

References:

[1] S. Feng, X. Zhang, D. Shi, Z. Wang, Front Chem. Sci. Eng., 15, 221-237 (2021)

[2] L.C. Agra, M.P. Lins, P.S. Marques, S. Smaniotto, C.B. de Melo, V. Lagente, E. Barreto., Eur. J. Pharmacol. 780, 232-242 (2016)

[3] O. Karagiaridi, M.B. Lalonde, W. Bury, A.A. Sarjeant, O.K. Farha, J.T. Hupp, J. Am. Chem. Soc. 134, 18790-18796 (2012)

Development and characterization of chitosan nanoparticles loaded with different aminoglycoside antibiotics

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Chitosan is a biodegradable and biocompatible polymer that has large potential for biomedical applications, particularly in the nanomedicine field, for the development of drug delivery systems. Besides other applications, chitosan nanoparticles (CS NPs) can be used to encapsulate drugs, promoting sustained release, improving drug bioavailability and stability [1].

This study consisted of developing and characterizing CS NPs loaded with two broad spectrum aminoglycosides, gentamicin or tobramycin. Chitosan-gentamicin (CS-G NPs) and chitosan-tobramycin (CS-T NPs) nanoparticles were obtained by dropwise addition of an aqueous solution of tripolyphosphate to a mixture containing chitosan and antibiotic at 60% of the chitosan weight, under magnetic stirring. The systems were centrifugated at 3500 rpm to remove agglomerates. The release behaviour was performed in PBS at 37ºC, and the antibacterial activity against gram+ and gram- strains was assessed by Kirby-Bauer tests.

The results showed that CS-G NPs and CS-T NPs had average sizes ranging from 120-160 nm and 130-160 nm, and encapsulation efficiencies from 40-75% and 60-75%, respectively. Polydispersity index was around 0.2 for both systems. In addition, they were both found to be antibacterial against the bacteria tested. After a continuous release for 2 hours, the kinetics seemed to depend on the encapsulated antibiotic. Indeed, while CS-G NPs released a maximum of 70% of the incorporated antibiotic, CS-T NPs released only 55%. The higher retention of tobramycin may result in a more controlled and prolonged release system compared to CS-G-NPs.

In conclusion, CS NPs encapsulating different aminoglycosides were successfully obtained through a simple approach, which has not yet been fully explored in literature. CS-T NPs can be considered promising systems for long-term sustained release.

Acknowledgments

Biopark, Prati-Donaduzzi, NSERC, Carmen and Luiz Donaduzzi.

Reference[1]

Bashir S.M. et al. Materials 2022, 15, 6521

Development of gold nanorods associated with microvesicles and Brazilian red propolis and their application for photothermal prostate cancer therapy

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Prostate Cancer (PCa) is one of the most commonly diagnosed malignant diseases in men around the world. Even with recent advances in the treatment of PCa, 40% of patients with local tumors develop metastasis [1]. The application of gold nanorods (AuNR) associated with Brazilian red propolis (BRP) can be an effective therapeutic alternative in the treatment of CaP, by associating chemotherapy with phototherapy. BRP is known for its anti-inflammatory and antitumor properties. The aims of this work were the development of a formulation composed of AuNR associated with extracellular vesicles (EVs) and BRP and their biological evaluation in prostate cancer cells. AuNRs were prepared by seed formation method and functionalized with thiol polyethylene glycol (PEG-SH). For association of AuNR-PEG with EVs and BRP a sonication method was used. The final dispersion showedtwo absorbance bands measured by UV-Vis, the largest one at 760 nm. AuNR-PEG-EV-BRPwere characterized by Transmission Electron Microscopy showing an aspect ratio of 3.2 ± 0.8. Hydrodynamic diameter (163.2 ± 21.8 nm) was obtained by Dynamic Light Scattering and Nanoparticle Tracking Analysis. Zeta potential value was negative. Biological studies were performed using prostate normal (PNT-2) and prostate cancer cells (PC-3 and LNCaP). The viability assays were carried out in the absence and presence of NIR light. The results demonstrated an enhanced antitumor effect after the system was irradiated using NIR laser (808 nm). Moreover, viability assays in normal cells showed low cytotoxicity. These results suggest the potential application of AuNR-PEG-EV-BRP in cancer therapy.

DOPED CARBON NANOPARTICLES AS AGENTS FOR ANTI-CANCER THERAPY

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The search for new therapeutic approaches for cancer is important since current resources are still insufficient. One option is Photodynamic Therapy (PDT), which generally acts by leading to the formation of reactive oxygen species due to the transfer of energy to photosensitizing molecules (PS) upon exposure to a beam of light. However, the use of conventional FS agents is limited due to the low penetration of light and the low diffusion of these agents to tumor cells. In this context, nanotechnology emerges as a potential solution to issues of improving the efficiency of PDT, where carbon nanoparticles (NPs) have been applied in several recent types of research. The present work sought to investigate the behavior and stability in the biological environment of carbon NPs doped with Nitrogen (N-GOQD), Boron (B-GOQD), and Phosphorus (P-CND), as well as their cytotoxic activity in neoplastic cells, to that these can be applied and/or modified for the application in GT. The characterization of the particles by transmission electron microscopy image indicated that these particles present little variability in their size and shape. To understand the behavior of NPs in a biological environment, assays were carried out to assess in vitro cytotoxicity (MTT assay) using neoplastic cells of the macrophage lineage J774.G8. In 24h of exposure and with increasing concentration, B-GOQD has the highest cytotoxic potential. When compared, N-GOQD and P-CND did not show significant cytotoxicity against the J774.G8 strain. Preliminary tests for the evaluation of the uptake of NPs by the cells show that, in 4h of exposure, about 10% of the concentration of P-CND particles are captured or deposited on the surface of the cells. The characterizations carried out on the carbon NPs applied in the present work bring important information about their physicochemical and biocompatibility properties, however, there is still a need for additional tests to obtain a good photosensitizing agent

Electrically conductive polyaniline-derived nanofibers as active interface for biomedical applications

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Polyaniline and poly(lactic acid) (PLA) blends are considered a promising combination for biomedical applications involving electrical challenges and impedance determination. Nonetheless synthesizing these polymer blends require the use of novel strategies [1]. In this work, polyaniline (PANI) and its derivatives poly(o-methoxyaniline) (POMA) and poly(o-ethoxyaniline) (POEA) were chemically synthed using an ultrasonic method. The synthed polymers were doped with p-toluene sulfonic acid and electrospun as blends with PLA, originating fibers of approximately 100 nm in diameter for all groups. Physicochemical and thermal properties were assessed by TCA and DSC analyses. The structural features of each membrane composite were also compared by XRD, FTIR, SEM and ElectrochemicalImpedance Spectroscopy (EIS) to determine their charge transport behaviors. The biocompatibility of the nanofibers was evaluated by in vitro tests with the human triple-negative breast adenocarcinoma MDA-MB-231 cell line and cytotoxicity assays using SYTOX™ Green dye for live/dead cells quantification and flow cytometry analysis. Taking all together, PLA/POMA membranes demonstrated the best electrical properties, with adequatein vitro biocompatibility. All tested nanofibers preserved ~ 90% of cell viability after 48 h incubation with MDA-MB-231 at 37°C/5% CO2, similar to controls. Our results present thePLA/POMA blend as a promising matrix with broad medical potential, as for the use in liveimpedance measurement strategies and/or for specific electric potential protocols for therapeutic purposes [2].

Acknowledgements:

The authors would like to thank to the Unidade Multiusuário de Citometria - CCS/UFRJ for the providing infrastructure and BD LSRFortessa™ equipment use. For the supported grants from CNPq, CAPES and FAPERJ.

References:

[1] C. Eiras et al., Química Nova 30, 1158-1162 (2007)

[2] N. Alegret, A. Dominguez-Alfaro and D. Mecerreyes, Biomacromolecules 20(1), 73-89 (2018)

Electrosynthesis of silver sulfide quantum dots for nanotheranotics

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Nanotheranostics is a powerful combination of diagnosis and imaging through nanoparticle systems. In this work, gluthatione capped silver sulfide quantum dots (Ag_2S-GSH QDs) are studied with the objective of promoting the diagnosis of tumors by fluorescence and treatment by photothermia. Ag_2S QDs were synthed via electrochemistry using a cavity cell,[1] where the optical properties of the nanocrystals were modulated by changing the molar ratio $Ag^+/HS^-$and the heat treatment. The nanocrystals present optical emission in the near infrared region, at wavelengths ranging from λem = 735 - 916 nm for the proportions1:1 and 1:4 (Ag^+/HS^-), respectively. The nanocrystals had an average diameter ranging from 4.2 nm to 5.5 nm, with a monoclinic crystalline structure [2]. When Ag_2S QDs were irradiated with laser in the near infrared region (λexc = 785 nm) they showed a photothermal response, ranging from 27 ºC for Ag_2S (6:1 ratio) to a maximum of 39 ºC (1:1 ratio) when covered with zinc selenide seeds (Ag_2S@ZnSe). MTT tests for HT29 cell line were performed, showing no toxicity, according to ISO 10993-5:2009. (Biological evaluationof medical devices: Tests for in vitro cytotoxicity i.e., cell viability >70%). The proposed system represents a green path to produce promising nanomaterials for application in nanotheranotics, through the electrochemical route and compositional engineering, combining a lower environmental impact with increased system efficiency.

[1] Areias, M. C. C.; Navarro, M. Electrochimica Acta, vol 53, 6477-6483, 2008
[2] Sadanaga, R.; Sueno, S. Mineralogical Journal, vol 5, 124-148, 1967
[3] Ding, C.; Huang, Y. Advanced Materials, vol 2007768, 1-29, 2021

Acknowledgements: CEMENE, CETENE, CAPES, UFPE/PGMTR, CNPq, MCTI.

Evaluation of synthesis conditions and luminescence properties of fluoroperovskite nanoparticles with application in photodynamic therapy

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Photodynamic therapy (PDT) is an alternative treatment for cancer. However, it is limited to tumors accessible with a laser in the UV-Visible region (active region of photosensitizers (PS)), the molecule responsible for cell death after its activation with a light source). Still, this energy range has low penetration into biological tissues. To extend the PDT to non-superficial tumors, we propose the development of fluoro-perovskite nanoparticles with upconversion (UC) properties, which will allow access to the PS (Aminolevulinic acid) to the inner tumor cells. UC enables the transformation of photons from the IR region (greater penetration of biological tissue) to the active region of the photosensitizer (405 and 635 nm), reaching non-superficial tumors. The UC property is achieved by incorporating dopants (rare earth and transition metals) into the nanoparticles. We investigated the solvothermal synthesis of $KMgF_3$nanoparticles through a rational design. Using statistical tools, we evaluated the influence of parameters such as precursor concentration, time, temperature, and dopant level obtaining nanoparticles with optimal properties for PDT. The statistical analysis yielded that the variable temperature and the interaction of temperature with the concentration of $MgCl_2$have a significant effect on the particle size, and that 14 of the experimental conditions did not present significant differences in particle size for a significance level of 0.05. Additionally, $KMgF_3$nanoparticles were doped with different concentrations of Nd^{3+}, Mn^{2+}, and Nd^{3+}/Mn^{2+}. The nanoparticles' optical properties were evaluated, confirming the upconversion process for samples with the highest concentration of $Nd^{3+}$and Mn^{2+}. Advances in this topic allowed us to move forward to the next steps involving functionalization and bioassays to evaluate the potential application of $KMgF_3:Nd^{+3}/Yb^{3+}/Mn^{2+}$in PDT.

Exploring the Role of Glycocalyx and Protein Corona inthe Cellular Uptake of Silica Nanoparticles

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Engineered nanoparticles (NPs) have remarkable potential for diverse biomedical applications. Over the last few decades, numerous studies have focused on characterizing and understanding the biointeractions of NPs with cells, especially factors that affect cellular internalization of particles. However, predicting or explaining specific outcomes remains challenging due to the complex nature of NP-cell interactions and endocytic processes. One the one hand, the formation of an adsorbed protein corona around NPs is a critical factor that influences NP-cell interactions and endocytosis. In addition, when NPs approach the cell surface, they first encounter the glycocalyx (GCX), a negatively charged glycosaminoglycan (GAG)-rich layer attached to the plasma membrane. Recent studies have suggested that the GCX may play an important role in regulating endocytosis, as it can either hinder or facilitate the process, depending on both the NPs'' and GCX’ s characteristics. However, interactions between the protein corona and the GCX and their impact on NP uptake remain poorly understood. Recently, our group investigated how the GCX modulates the cell uptake of charged NPs in the absence of a protein corona. In the present study, silica NPs were covered by well-defined coronas of albumin (BSA), cationized albumin (BSA+) and fetal bovine serum (FBS). The NPs were exposed to wild-type and GAG-mutated CHO cells and uptake was quantified by flow cytometry. Additional cell uptake experiments were performed using wild-type cells in the presence of glycolytic enzymes or chemical/pharmacological inhibitors. Furthermore, biophysical measurements were performed to understand the interactions between the protein coronas and free GAGs. We found that free GAGs bound not only to BSA+ coronas, as expected, but also to BSA and FBS coronas despite their negative zeta potentials. Surprisingly, CHO cells containing intact GAGs showed enhanced NP uptake for all coronas.

Illuminating the Biological World through Infrared Radiation at IMBUIA Beamline on Sirius

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IMBUIA Beamline (Microspectroscopy and Infrared Imaging) is an advanced facility dedicated to the study of materials at molecular and nanoscale levels. With its high brightness, it offers synchrotron-based infrared spectroscopy and imaging techniques, allowing researchers to explore the molecular and vibrational properties of samples with exceptional sensitivity and spatial resolution. The use of infrared radiation (IR) in the investigation of biological systems has proven to be a valuable tool to analyze the molecular composition, structure and dynamics of samples without the need for chemical markers. For example, it can detect DNA damage in fungi exposed to volatile organic compounds, using the natural molecular vibrations of the samples. IR spectral microscopy provides chemically specific information with spatial resolution, allowing researchers to map the distribution of specific biomolecules within tissues and cells. Infrared imaging has proven particularly useful in cellular research as it facilitates the characterization of local chemical variations and the identification of specific components within complex systems. By integrating spatial and spectral information, the composition and distribution of materials in biological, geological and materials science research is revealed. The versatility of the IMBUIA Beamline, with its synchrotron-based infrared microspectroscopy and imaging capabilities, offers powerful opportunities to delve into the molecular world and gain a deeper understanding of material properties. The application of IR in the study of biological systems offers benefits such as characterization of biomolecules, spatial imaging, probing interactions and changes and dynamic analysis without relying on chemical markers. These applications will significantly advance our understanding of the biological world and drive technological progress in infrared spectroscopy.

Acknowledgments: CNPEM - LNLS.

[1] Freitas et al. 24 (3),1430-1453, 2022.

Interaction Mechanisms of Carbohydrate Functionalized Silica-Based Nanoparticles and Bacteria: A Nano-FTIR Synchrotron Study

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Nanotechnology has been increasingly investigated as a promising strategy to address the growing challenge of antimicrobial resistance [1]. Nevertheless, the current understanding of the interaction mechanisms between nanoparticles (NPs) and bacteria remains limited, largely due to the spatial resolution constraints of conventional surface characterization techniques, as Fourier transform infrared (FTIR) and Raman spectroscopies [1-2]. The aim of this work is to monitor the surface interaction between *Escherichia coli* (*E. coli*) and silica-based NPs (SiO_2NPs) coated with carbohydrates, using a cutting-edge approach that combines Scanning Near-field Optical Microscopy (s-SNOM) with broadband synchrotron infrared nanospectroscopy (nano-FTIR). To enhance the interaction between the NPs and the bacterial membrane at the nanoscale, SiO_2NPs were coated with the carbohydrates mannose, maltose or trehalose, since Gram-negative bacteria are known to recognize and interact with glycans [3]. Physical-chemical characterization techniques were performed in order to confirm the presence of carbohydrates, and the interaction between NP's and bacteria were studied by biological *in vitro* assays and scanning electron microscopy. Moreover, the resulted carbohydrate-functionalized SiO_2NPs were evaluated for colloidal stability in several media. This work presents a unique approach for studying the nano-bio interface using the Nano-FTIR technique, which enables the chemical and morphological analysis of a single cell''s surface with a remarkable spatial resolution of 30 nm. This advanced capability was made possible by the Imbuia-Nano beamline at Sirius in Campinas.

References:
1 Capeletti, L.B., et al. Advanced Functional Materials. 2019, 29, 1-11
2 Szostak, R.; et al. Science Advances. 2019, 5, 1-7
3 Day, C.J.; et al. PNAS. 2015, 112, 1-10

Acknowledgment: We thank FAPESP (process number: 21/11858-2), CNPEM (LNLS, LNNano and LNBio), and UNICAMP for all financial support along this work.

Magnetic plasmonic $NiFe_2O_4$–silver nanoparticles: a potential SERS biosensor for newborn screening tests

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Multifunctional nanosystems that combine magnetic and plasmonic properties are a promising approach for the development of new testing diagnosis devices due to the straightforward method for removal of the analyte from a complex matrix, in addition to the high sensitivity provided by surface-enhanced Raman scattering (SERS) [1-2]. In order to develop an approach for detecting diseases such as congenital adrenal hyperplasia, which is included in the routine screening of newborns, nickel ferrites ($NiFe_2O_4$) were synthed using the reverse micellar microemulsion method, following by their functionalization with cysteamine for being decorated with silver nanoparticles (AgNPs). Furthermore, the diffraction pattern shows the maximum profile of AgNPs, where an increase in peak intensity (111) was observed due to the high concentration of AgNPs on nickel ferrite surface, corroborating with SEM which shows a continuous layer of Ag on the nickel ferrite surface. According to VSM characterizations $NiFe_2O_4$ exhibited low coercivity, moderate saturation magnetization and low hysteresis losses, being classified in the soft ferrite class. To determine the chemical environment and oxidation state of the silver atoms on nickel ferrites, the samples were characterized by XPS at the central levels of 367.86 and 373.70 eV, assigned to Ag $3d_{5/2}$ and Ag $3d_{3/2}$, respectively. The potential of $NiFe_2O_4$–AgNP as a SERS substrate was demonstrated with methylene blue (MB) as target molecule. Therefore, magnetic plasmonic $NiFe_2O_4$–AgNP are promising for the development of biosensing tests as an alternative for routine analysis.

Acknowledgements: CNPq, CAPES, FAPERGS, PPGCiMat, PPGQ, FIOCRUZ, UFRGS

[1] Wang, C., Meloni, M. M., Wu, X., Zhuo, M., He, T., Wang, J. Dong, P. (2019). AIP advances, 9 (1), 010701 (2019).

[2] Perveen, S., Hafeez, S., Rashid, S., Asad, S. U., Khan, M. Z., Azad, F. Materials Chemistry and Physics, 127303. (2023)

Mesoporous silica biomimetic nanoparticles for lung cancer therapy

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Nanotechnology is an alternative to resolve the gaps in conventional treatments through designing drug delivery systems. The mesoporous silica nanoparticles (MSN) are proposed as a potential drug delivery for targeted non-small cell lung cancer therapy. These nanoparticles were synthesized by a co-condensation method and optimized employing physical-chemical treatments to use as nanocarriers of the chemotherapeutics drug carboplatin. The MSN showed different sizes and zeta potential depending on each treatment. Calcined MSN nanoparticles showed a diameter of ca. 88 nm, as observed by TEM microscopy and Pζ of -15.4 mV. MSNs functionalized with (3-aminopropyl)triethoxysilane (APTES) after-synthesis or treated with reflux, registered a diameter between 114 nm with Pζ of 35.2 mV, and 172 nm with a Pζ of 52.3 mV, respectively. The surface chemistry of MSN plays a vital role in the load and release drug, which was studied via MicroRaman and FTIR spectroscopy. In all MSNs spectra were observed transverse optical modes referring to the siloxane bond at 800 cm^{-1}(ν_s(Si-O-Si)$_{TO}$) and 1085 cm^{-1}(ν_{as}(Si-O-Si)$_{TO}$). The conversion between surface siloxanes and hydrogen-bonded silanols were observed for non-calcined nanoparticles, along APTES functionalized assigned same mode of the NH_2-CH_2-CH_2segment (shoulder at 1448 cm^{-1}) and bands observed for high Raman shifts corresponding to the connections between the $NH_2$and Si(-O-)$_3$. For the FTIR spectra, the vibrational modes of the ν(Si-O-Si) band were found at 1061 $cm^{-1}$and 1223 cm^{-1}, respectively. The Amine group was identified in the same region as Raman spectra for N-H stretching and bending mode. Non-calcined MSNs have a hydrophilic surface functionalized with APTES, which is more advantageous for drug delivery systems.

Acknowledgments:
The funding agency: to CNPQ DTI-A 383239/2022-2

Protein corona: An investigation of the interactions between serum albumin and silica nanoparticles

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Nanoparticles are promising drug delivery systems for many pathological conditions [1]. However, low targeting efficiency in vivo has been registered. A potential cause for this is that proteins spontaneously bind to the surface of nanoparticles when in biological fluids [2], forming a protein corona that affects the nanomaterial's properties, masking engineered functionality [3]. Therefore, to contribute to the development of more effective nanocarrier platforms, it is necessary to comprehend the formation of the protein corona and its consequences. Thus, this study is based on the investigation of the adsorption of human and bovine serum albumin (HSA and BSA) proteins onto silica nanoparticles of different s. Previous experiments using ultra-violet circular dichroism indicated that BSA may undergo structural rearrangement when bound to silica nanoparticles of approximately 170 nm. In order to explore the chemistry of the protein layer and evaluate possible conformational changes without the need for chemical labeling, two other techniques will be used: micro infrared spectroscopy with a microfluidics approach, and synchrotron infrared nano-spectroscopy.

References:

[1] CAPELETTI, L. B. et al. Silica Nanoparticle Applications in the Biomedical Field. Smart Nanoparticles for Biomedicine, Elsevier. 2018.

[2] MIRSHAFIEE, V. et al. Impact of protein pre-coating on the protein corona composition and nanoparticle cellular uptake. Biomaterials, Elsevier BV. 2016.

[3] AFFONSO DE OLIVEIRA, J. F. et al. Dual Functionalization of Nanoparticles for Generating Corona-Free and Noncytotoxic Silica Nanoparticles. ACS Applied Materials amp. Interfaces American Chemical Society (ACS). 2018.

Real-Time Detection of Magnetic Nanoparticle Distribution Using Optically Pumped Magnetometers: A Pilot Study in Rats.

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Magnetic nanoparticles (MNPs) have unique properties that make them useful for biomedical and clinical research. They can be monitored using magnetic fields and detectors such as optically pumped magnetometers (OPMs). The National Institute of Standards and Technology (NIST) has developed chip-scale OPM sensors with sensitivities below 200 fT/Hz, which have been used in fetal magnetocardiography and magnetoencephalography studies. In a pilot study, MNPs were administered to male Wistar rats intravenously, and their distribution was monitored in real-time using a gradiometric array of QuSpin Total-Field Magnetometer (QTFM Gen-2) OPMs. The OPMs recordings allowed for the assessment of the arrival time of particles at the heart, their accumulation and subsequent emptying from the organ, and the determination of their circulation time. The results demonstrate the suitability of using OPM sensors for real-time detection of MNPs distribution and suggest a new possibility for evaluating their biodistribution. Future in vivo animal experiments are expected to use quantitative magnetic images to determine the passage of MNPs through the heart and hepatic uptake by the liver without external magnetization and without the need for a shielded room. Acknowledgement Fapesp process 2021/06405-9.

siRNA controlled release from porous silicon nanoparticles coated with hyaluronic acid

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The RNA delivery is emerging in the last century as a new class of therapeutic for a wide range of diseases. Delivering RNA is a very interesting strategy for cancer therapy and rare diseases,[1]but remains a great challenge because of its low cell uptake, instability in blood. For this reason, the use of lipidic nanoparticles has been recently witness as the solution for COVID 19 and the use of other type of nanocarriers is a promising approach for the RNA delivery and its controlled concentration over time. The problem is: what type of nanoparticles can be biocompatible, degradable and able to load enough RNA for therapeutic efficacy? Porous silicon nanostructures are of particular interest because of they fulfill all the requirements to allow in vivo applications, and they are easy to functionalize. The high porosity of these structure also allow a large amount of RNA entrapment and the structures prevent the degradation of the biomolecule and allow the specific drug release ina targeted area.[2]However, due to the fast degradability of the silicon, a coating with a polymeric layer is necessary to increase its stability and control the time of release. Here, we report a strategy to load a high amount of RNA and, to avoid its early release, the use of hyaluronic acid (HA) wrapped around the particles. The presence of HA also helps to be selective towards the tumor environment due to the presence of overexpressed CD44 receptors specific for the polymer. Also the release of the RNA is achieved by the HA degradation promoted by an enzyme called hyaluronidase.[3]In this work, is achieved a loading higher than 50% in weight thanks to the use of a calcium silicate reaction and we demonstrate the difference in the time release of RNA between nanoparticles coated and uncoated with HA.

THE PROCESSING OF CELL MEMBRANES AS ACTIVE MATERIALS FOR NANOCARRIERS FABRICATION AND THEIR APPLICATION IN PHOTO/CHEMO THERAPIES AGAINST CANCER

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Recent advances in nanoengineering have allowed the isolation of both cell membranes (CM) and extacellular vesicles (EVs) to be used as efficient biomimetic nanosystems to deliver organic and inorganic molecules for cancer therapy. In this study we report the development of EVs and cell membrane-based nanocarriers encapsulating nanoparticles and anticancer drugs for both cancer photothermal and chemotherapies, respectively. Our results reveal a higher tendency of the EV-coated nanorods to interact with macrophages yet both EV- and cell membrane-coated nanorods were internalized in the metastatic breast cancer cells. Additionally, highly stable nanoparticles containing Gemcitabine (GEM), Paclitaxel (PTX), and Temozolomide (TMZ) were obtained using the major components isolated from tumor cell membranes. The nanoparticles containing GEM or PTX (NP-GEM-PTX) induced higher cytotoxic effects on pancreatic PANC-1 cells, in comparison to the use of pure GEM+PTX, indicating that membrane-covered nanoparticle favors the targeting and interaction with tumor cells. The biomimetic NP-TMZ system was built with isolated cell membrane from a U251 glioblastoma cell line and showed a greater affinity to U251 cells compared to other glioblastoma cell lines. The internalization of MNPs-TMZ in U251 cells was almost 2 times greater compared to U87 cells, and nearly 4 times greater compared to HCB151 cells. The use of EV- and cell membrane-coated nanoparticles opens up new possibilities regarding the development of efficient biomimetic nanosystems for cancer therapy and brings benefits to the field of personalized medicines.

2D and 3D spectroscopic studies, cellular uptake, and cytotoxicity assays of zinc phenyl-thio-phthalocyanine polymeric nanocapsules

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Polymeric nanocapsules (NCs) are a drug delivery system consisting of biodegradable polymers and an oily core that acts as a reservoir and have attractive characteristics such as greater solubility, absorption, bioavailability of drugs and cellular specificity. The present work aims to determine the absorption spectrum (UV-Vis), fluorescence emission (2D and 3D) of free ZnS4pc and associated with polymeric nanocapsules (NC/ZnS_4Pc). In Vitro Biocompatibility and cellular uptake of NC/ZnS_4Pc in two types of normal human cell lines (HdFn and HaCaT). The NCs were prepared by the interfacial deposition method with anionic surfactant Kolliphor 188, PLGA copolymer (50:50) (poly (lactic-co-glycolic acid) (50:50)), soy lecithin, soy oil and ZnS_4Pc. To determine the absorption spectrum of free and associated ZnS_4Pc in nanocapsules in organic medium (acetonitrile and DMSO), spectroscopy studies (UV/Vis) were performed. Spectrofluorimetric analyzes were performed in 2D and 3D mode. Cytotoxicity and cell uptake study was conducted using different concentrations of NC/ZnS_4Pc in HdFn and HaCaT cell lines using the resazurin colorimetric method. The absorption and fluorescence emission spectrum (2D and 3D) of ZnS_4Pc and NC''s/ZnS_4Pc in organic medium showed similar profiles. In vitro cytotoxicity observed that NC/ZnS_4pc concentrations of 0.5, 1.0 and 2.0 µmol.L^{-1}showed no toxicity compared to the control for both cell lines tested. The concentration of NC/ZnS_4pc that demonstrated the best profile in the cell uptake assay was 2 µmol.$L^{-1}$for both cell lines tested. Photophysical characteristics at steady state did not change when carrying ZnS_4Pc in nanocapsules compared to free ZnS_4Pc. Therefore, the results obtained so far were considered promising for the next steps in 3D biological assays. This work was supported by FAPESP (The São Paulo Research Foundation), Grants M.A.F. nº 2020/09396-8 and CNPq Universal Project Grants F.L.P nº 404416/2021-7.

Acute Oral Toxicity Evaluation of CZIS/ZnS Quaternary Quantum Dots and Potential use as a Fluorescent Probe

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Abstract: In vivo imaging is essential to identify cancer and increase patient survival. As an alternative to conventional techniques for monitoring tumors, in vivo imaging fluorescence has significant advantages, such as recognition of specific targets. As an attractive alternative to traditional fluorescent probes, quantum dots (QDs) have several advantages for biological analysis, such as, high quantum yields and excellent photostability [1]. There are countless possibilities for applying QDs in biological environments, but there is a lack of information regarding the effects generated in the short and long term. In this work, an acute oral toxicity test was performed for QDs CZIS/ZnS solubilized in phosphate buffer, according to Guideline 423[2]. The animals were divided into 3 groups formed by vehicle 300 mg/kg and 2000 mg/kg. They were evaluated for 14 days and then euthanized when their organs and blood were collected for further analysis. The highest dose showed 100% lethality, however, the 300 mg/kg dose showed no immediate effect, and no mortality. No side effects were observed in white and red blood series counts ($p>0.05$), as well as in relative organ weight ($p>0.05$), and estrous cycle duration ($p>0.05$). Therefore, CZIS/ZnS can be considered in category 4, according to the Globally Harmonized System of Classification and Labeling of Chemicals (GSH), with a Median Lethal Dose (LD50) >300-2000 mg/kg, revealing its very low toxicity. Altogether, these findings indicate that QDs CZIS/ZnS showed great potential to be used as a cellular probe and more studies has been done to validate its application.

Acknowledgments

This work was supported by CNPq, FINEP, CAPES and FAPEMIG.

References

[1] Santos, H. D. A. et al. Ultrafast photochemistry produces superbright short-wave infrared dots for low-dose in vivo imaging. Nature Communications, 11, 2933, 2020.
[2] OECD. Test No. 423: Acute Oral toxicity – Acute Toxic Class Method. Oecd Guidel. Test. Chem. 1-14, 2002.

Advancing Newborn Screening Tests for Cystic Fibrosis: Affordable Screen-printed Sensor Based on Polypyrrole-AgCl nanocomposites

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Cystic fibrosis (CF) is a genetic disorder that affects the respiratory, digestive, and reproductive systems. Early diagnosis and treatment are critical for improving the quality of life and survival rates of CF patients. To facilitate early diagnosis, newborn screening programs have been implemented in many countries, which involve the collection of dried blood spots from newborns and subsequent analysis. However, current screening methods are expensive, invasive, time-consuming, and require specialized equipment and trained personnel, which limit their accessibility to remote areas where the neonatal screening program has no coverage. Currently, the most common strategy for CF diagnosis involves the determination of chloride concentration in sweat, as in case of a positive diagnosis the chloride concentration is over 60 mmol L-1.[1]

In this context, we developed polypyrrole-AgCl nanocomposites (PPy-AgCl-NC) which showed great potential for the development of electrochemical sensor for chloride. We investigated the chemical polymerization mechanism of pyrrole, in the presence of methyl orange, FeCl3 and different concentrations of silver nitrate, that allowed insitu formation of AgCl nanoparticles inside and outside of the polypyrrole structure depending on the concentration. The PPy-AgCl-NCs were characterized by scanning and transmission electron microscopies, showing the presence of polypyrrole nanostructures, and AgCl nanocomposites, respectively. When applied to chloride ions detection, the sensor demonstrated a remarkable faradaic response over a wide concentration range from 10 to 400 mmol L-1. Cyclic voltammetry was carried out in [Fe(CN)6]3-/4- solution to characterize the electrochemical parameters. This new approach holds promise for the diagnosis of CF and offers advantages in terms of low cost and reproducibility.

Acknowledgements:

CNPq, CAPES, PPGCiMat, FIOCRUZ

References:

[1] Toh HS, Batchelor-Mcauley C, Tschulik K, Compton RG, 15., 4292-7 (2013).

A new smart drug delivery nanosystem based on magnetic nanospheres coated with biopolymer is prepared as a nanocarrier for curcumin in the treatment of Alzheimer's disease

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The present study describes the synthesis, characterization (SEM, FT-IR, TGA, zeta potential, point of zero charge (pHPZC), and XRD), and in vitro study (delivery kinetics, membrane permeation, solubility isotherm, and toxicity) of a new smart nanocarrier based on magnetic nano-spheres (Fe_3O_4) functionalized with chitosan (CS) as a controlled release vehicle for CUR - (Fe_3O_4@CS-CUR) - for AD therapy. Characterization results showed that the synthesis and functionalization of the material occurred satisfactorily, as evidenced by FT-IR and TGA analysis. From SEM data, it was observed that the nanomaterial had an amorphous, irregular, and heterogeneous surface, containing iron nanoparticles impregnated on the surface of the chitosan polymer matrix, as evidenced by characteristic XRD peaks. The nanomaterial had an average size of 169 nm with a zeta potential of −30.7 mV. Fe_3O_4@CS-CUR presented a pHpzc at pH 8.0. The release study showed that at pH 7.4, the release of curcumin occurred in a controlled manner by the nanocarrier. Kinetic study indicated a second-order profile (R^2 = 0.96) for the delivering of CUR. Membrane permeation study on synthetic cellulose membrane (which simulated the blood-brain barrier) showed that Fe_3O_4@CS-CUR had satisfactory permeation, delivering CUR in a controlled manner. In the toxicity study, low toxic effects of the prepared nanocarrier were observed, with an EC50 (effective inhibitory concentration of 50%) of 554 µg mL-1. These data show that a new nanocarrier with potential application in AD treatment and low toxicity to cells has been successfully prepared.

Acknowledgements:FAPEMIG (APQ-01786-22; RED-00144-22); CNPq (405828/2022-5) and CAPESReferences[1] M. M. Wen et al. Journal of Controlled Release vol. 245, 95-107 (2017)[2] S. D. Voulgaropoulou et al. Brain Research vol. 1725, 146476 (2019)[3] M. Ray et al. Nanomedicine vol. 12, 941-952 (2017)

Antibacterial alternatives for the use of Nile tilapia skin impregnated with green AgNPs and ZnO-doped green AgNPs

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The selection of antibiotic-resistant bacteria requires the urgent development of alternatives to conventional antibiotics. With regard to skin lesions, several materials and treatments for injuries are already available. As a part of innovative therapeutic approaches against infections, Nile tilapia skin-based dressings present biological similar composition to human skin [1] and the reinforcement given by impregnation with silver nanoparticles due to their considerable antibacterial activity [2]. Herein, silver nanoparticles were reduced by the use of the extract of Ziziphus joazeiro, in association with zinc oxide, applied as an additive for the antibacterial activity of silver. The synthed silver nanoparticles and complexes of Ag and ZnO were characterized in terms of , zeta-potential and scanning electron microscopy as particles and after incorporation in Tilapia skin. The antibacterial activity was evaluated against Staphylococcus aureus and Escherichia coli with assays of zones of inhibition, kill time and biofilm inhibition. Reduction in the minimal bactericidal concentration was observed for samples prepared at increasing concentrations of zinc oxide with a more evident variation for E. coli. These results were confirmed by inhibition halo zones that were larger for complexes of Ag+ZnO against E. coli. As a consequence, the best kinetics of bacterial inhibition was observed for complexes (Ag+ZnO) against E. coli (elimination of organisms after 1 h) indicating the synergism of components and an effective action against Gram-negative species due to the thin peptidoglycan cell wall relative to the Gram-positive species.

Acknowledgments: FACEPE, CAPES, CNPq and FINEP.

References:

[1] E. M. L. Junior; N. S. Picollo; M. J. B. de Miranda; W. L. C. Ribeiro; A. P. N. N. Alves; G. E. Ferreira. Rev Bras Queimaduras., v. 16, p. 10-17 (2017).
[2] M. L. GUIMARÃES; F. A. G da Silva; M. M. da Costa; H. P. de Oliveira. Applied Nanoscience, v. 10, p. 1073-1081 (2019)

Azithromycin reactivity on Ce2(MoO4)3/MWCNT/graphite-based composite electrode: an investigation in electrolytes formulated with CH3OH/H2O blends

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Azithromycin (AZT) is a semi-synthetic antibiotic derived from erythromycin, whose consumption of non-administered doses arouses serious side effects, including the emergence of more resistant bacteria [1]. AZT is also an endocrine disruptor, triggering toxicological effects in non-target organisms. Despite the need to monitor it in relevant samples, assays become limited due to its low solubility (0.1 mg mL-1) in purely aqueous solutions [2]. Little is known about AZT electrochemistry to develop more robust analytical methods, largely due to its structural complexity (15-membered ring with sugar residues attacked) and low interfacial reactivity of the semiconductors employed. In this work, we present data obtained with a composite electrode based on cerium (III) molybdate, multi-walled carbon nanotubes and graphite, here defined as Ce2(MoO4)3/MWCNT/graphite, which showed satisfactory performance to explore AZT oxidation in CH3OH/H2O blends. Semi-emirical simulations indicated that the CH3OH content (5-50%, v/v) alter the blend's dielectric constant, expose hydrogen bonding sites and favor drug solubility. The electrochemical reaction assisted by voltammetry and impedance was already clear on graphite electrode, using phosphate buffer (pH = 8.0; 10% methanol, v/v), but it was amplified and catalyzed by combining MWCNT and Ce2(MoO4)3, respectively, in the sensor composition. The results prospect the Ce2(MoO4)3/MWCNT/graphite composite as a promising material for electroanalytical studies involving AZT in complex matrices.

Acknowledgments:

CNPq, CAPES, Funcap and UFCA.

References:

1. Parnham MJ, Erakovic Haber V, Giamarellos-Bourboulis EJ, Perletti G, Verleden GM, Vos R. Azithromycin: mechanisms of action and their relevance for clinical applications. Pharmacol Ther. 2014

2. Aucamp, M., Odendaal, R., Liebenberg, W., Hamman, J. Amorphous azithromycin with improved aqueous solubility and intestinal membrane permeability. Drug Development and Industrial Pharmacy. 2014.

Bimodal Nanoplatforms Containing HydrophilicAgInTe2 quantum dots QDs and DOTA-like Gd- complexes for imaging diagnostic tools

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The search for potential bimodal nanoparticulate probes based on gadolinium (III) chelates and quantum dots has been increasing since the last decade, mainly for its potential as a revolutionary tool in medicine, for magnetic and optical imaging, overcoming the limitations of both techniques [1]. Among the luminescent nanoparticles, we have the class of ternary I-III-VI quantum dots, which are considered environmentally friendly materials, for not having heavy metals as constituents. In addition, they are materials with high absorption coefficient, low thermal conductivity, efficient energy convection and good photostability [2,3]. In this work, we used the hydrophilic ternary quantum dots based on silver, indium and tellurium (AgInTe2) stabilized with MSA. The objective of this study is the conjugation of thiolated DOTA gadolinium (III) complexes modified with different linkers (CYS, PEN and HIS) and ternary quantum dots, obtaining versatile bimodal nanoprobes that provide an optical-paramagnetic improvement and then can be evaluated as potential bimodal contrast agents for optical and magnetic resonance imaging techniques. The synthed nanoplatforms were characterized by ultraviolet and visible (UV-Vis) absorption and emission spectroscopies, relaxometry (20 and 60 MHz, 37ºC).

Thanks to UFPE, CNPq, FACEPE (IBPG), and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020) for the financial support.

References:

[1] M. I.A. Pereira e G., Pereira, Scientific reports, v. 9, p. 1-11, 2019.

[2] M. Sandroni e K. D. Wegner, ACS Energy Letters, v. 2, p. 1076-1088, 2017.

[3] C. I. L. Santos e J. C. A. Ferreira, Quim. Nova, v. 43, p. 813-822, 2020.

Bimodal nanoprobes containing hydrophilic CdTe quantum dots functionalized with glutathione and Gd-complexes for diagnostic

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There is a growing interest in the development of multimodal probesthat have a signal for more than one diagnostic imaging technique. Magnetic resonance imaging (MRI) is a non-invasive technique capable of distinguishing pathological from healthy tissues. However, in some cases, a high local concentration of contrast agents (CAs) is required to improve the observable contrast in images. To overcome this limitation, nanoparticles (NPs) are being associated withCAs, concentratinge several CAs molecules in a single NP, increasing the local concentration of the paramagnetic ion, without increasing the administered dose.[1] These CAs can be associated with NPs that have optical properties, such as quantum dots (QDs).[2] In this work, we intend to associate CdTe QDs with gadolinium complexes, combining the advantages of MRI and optical imaging. The QDs were prepared in water, using glutathione (GSH) as the stabilizer. The DOTA ligand was linked to Cysteamine (CIS), GSH, and L-Cysteine (CYS) and complexed with Gd3+, showing values of relaxivity and stability similar to the DOTA-Gd complex used clinically. TheQDs were conjugated with the thiolated DOTA-Gd complexes, obtaining the bimodal systems. Optical characterization indicated that the QDs remained stable and fluorescent, observing an increase in emission intensity after conjugation. The nanoprobes were characterized by relaxometry at 60 MHz (37 °C) and, 20 MHz (25 °C and 37 °C), obtaining longitudinal relaxivitiesper Gd3+ superiorto the CAs used clinically. Thus, the prepared nanoprobes have the potential to be used asCAs for MRI and optical imaging.

Acknowledgments:
Thanks to UFPE, CNPq and FACEPE (BIC-1254-1.06/22) and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020) for the financial support.

References:
[1] ALBUQUERQUE, et al. Topics in Current Chemistry, 379, 2021.
[2] PEREIRA, et al. Scientific reports, 2019.

Biocompatibility analysis of the Simonkolleite nanomaterial conjugated with bioactive compounds in Drosophila melanogaster

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Previous studies carried out by the group revealed that the synergism of simonkolleite (SM) and zinc oxide (ZnO) nanocrystals (NCs) proved to be biocompatible and with antitumor effects. However, the biological effects of SM NCs is still unknown. In order to evaluate the potentiation of the properties of these NCs, nanoconjugation with bioactive compounds is a tool that can be employed. For this purpose, Drosophila melanogaster is a well-established model organism that allows a rapid assessment of the possible effects of nanocrystals as it isa versatile and relatively simple model. Thus, the main objective of this work was to evaluate the biocompatibility of the nanoconjugation of bioactive compounds inSimonkolleite nanocrystals in the fly model. We evaluated the effects of the SM NCs, pureand conjugated with curcumin and quercetin wild type lineage Canton S, in which such substances were mixed in the culture medium at different concentrations. We have evaluated parameters such as pupation per day, total pupation, larval lethality, negativegeotaxis and lifespan of exposed animals. Our data indicates that bioactive compounds conjugated with nanocrystals, when evaluated separately, present biocompatibility. Surprisingly, the well known anti-inflammatory substance quercetin, when combined withNCs, shows toxicity at higher concentrations, possibly due to the exposure of regions of thenanocrystals that were unavailable in the original conformation. Nowadays, we are evaluating the effects of curcumin conjugation on NCs. We hypothe that it may potentiatethe effect of the nanocrystals. Normally, natural substances known for their beneficial effects are expected to improve biocompatibility, however, our results show the importanceof investigating the synergism of the combination of such compounds.

Biocompatibility of Gold doped TiO_2 Nanocrystals in *Drosophila melanogaster*

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Titanium dioxide (TiO_2) can be applied in different fields since they present high thermal stability and catalytic activity, being of great industrial and pharmaceutical interest. The incorporation of gold (Au) in the crystalline structure of TiO_2 nanocrystals can generate new and interesting properties. However, is necessary evaluate the possible side effects and toxicity of the nanocrystals pure and doped in biological systems. Due to their short life cycle, ease of handling and genomic similarity to humans, the ***Drosophila melanogaster in vivo*** model was used to investigate the toxicity induced by orally administered Au doped TiO_2 nanocrystals. The nanocrystals were synthesized and the toxicological test was evaluated on development by inserting L1 larvae (30 larvae/replicate, six replicates per concentration) into different concentrations of Au doped TiO_2 (0.5; 1.0; 2.0; 4.0 mg/ml) added to Drosophila standard medium. The larval and pupal lethality rates were determined, along with the lifespan of animals exposed to the substance. The location of nanocrystals will be observed through fluorescence microscopy to track the tissue localization at different stages of the animal development. Our results suggest biocompatibility at the analyzed concentrations as it did not cause significant increase in larval lethality and decrease in pupation and lifespan. However, further studies are still crucial to evaluate the possible adverse effects of using these nanocrystals.

BIOGENIC IRON NANOPARTICLES USING *Penicillium chrysogenum* AS A STABILIZING AGENT

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Iron nanoparticles obtained from the biogenic synthesis method are non-toxic to humans. They are more biocompatible when compared to their chemically synthesized counterparts, have low toxicity and can help to inhibit the growth of bacteria, fungi and other pathogens. Biogenic Iron (FeNPs) nanoparticles were synthesized using the microorganism ***Penicillium chrysogenum***as a stabilizing agent and Iron III Chloride (FeCl3) as a metal precursor with concentration variations of 1mM and 5mM. After the synthesis, the physical-chemical characterization was performed to obtain the size, polydispersion index (PDI) and zeta potential. Cell viability in the presence of nanoparticles was assessed using the MTT assay, and the comet assay assessed DNA damage in 3T3 cells. A disk diffusion test in agar evaluated the antimicrobial activity. The results showed nanoparticles with a hydrodynamic diameter of 122 ± 1.23 nm, polydispersion of 0.237 ± 0.006 and zeta potential of -16.63 ± 0.45 mV. Viability analysis showed IC50 at 35% concentration, with no indication of genotoxicity. Activity evaluation by disk diffusion and MIC tests showed that NPsFe did not have antimicrobial potential. When evaluating the effects of NPsFe on seed germination, no changes were observed concerning the control. The biogenic synthesis was viable, with NPsFe with favorable characteristics. However, further studies on the cytotoxicity and genotoxicity of NPsFe are needed.

Acknowledgements: Universidade de Sorocaba e Unesp de Sorocaba

References: [1] SOLIMAN, A. M. et al. Green Approach to Overcome the Resistance Pattern of Candida spp. Using Biosynthesized Silver Nanoparticles Fabricated by Penicillium chrysogenum F9. Biological Trace Element Research, v. 199, p. 800-811, 2021.

Biomimetic nanoparticles encapsulating paclitaxel for lung cancer therapy

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Chemotherapy is a well-established treatment modality for lung cancer, being paclitaxel (PTX) an FDA-approved drug of choice for non-small cell lung cancer (NSCLC). However, PTX shows severe side effects and high toxicity, besides the potential of resistance development over time [1]. Polymeric nanoparticles offer a promising solution for delivering PTX, due to their unique properties, such as high loading capacity and good biocompatibility [2]. The present study aimed at the synthesis and characterization of a nanosystem based on poly (lactic acid-co-glycolic acid) (PLGA) for carrying PTX, as an alternative for the treatment of NSCLC. The optimization of the synthesis of PTX-loaded PLGA NPs was performed and two different solvents (Dichloromethane (DCM) and Acetone) were tested for the organic phase, as well as two surfactants (Pluronic (PLU) and Polyvinyl Alcohol (PVA)) at different concentrations. The physicochemical characterization of NPs was evaluated using Dynamic Light Scattering (DLS) and Nanoparticle Tracking Analysis (NTA). The PTX-loaded NPs exhibited a diameter of ca. 160 nm ± 36 nm with a PDI of 0.297 and ZP -16.9mV ± 6mV, indicating the NPs electrical stability. NTA revealed an average size of 125.1±1.5 nm, corroborating the DLS measurements. The NPs concentration was 4.47x1012 particles/mL. Scanning electron microscope (SEM) images revealed spherical nanoparticles with diameters in the range of 150 nm. The optimized synthesis conditions allowed the fabrication of PTX-loaded NPs as candidate nanotherapeutics for NSCLC therapy.

Acknowledgments: CAPES and FAPESP.

References: [1] Gallego-Jara, Julia, et al. Molecules. Vol. 25,24 5986. 17 Dec. 2020, doi:10.3390/molecules25245986.

[2] Mukherjee, Anubhab, et al. Cancers vol. 11,5 597. 29 Apr. 2019, doi:10.3390/cancers11050597.

Biomimetic nanovesicles composed of hybrid lung adenocarcinoma (A549) and lymphoblasts (Jurkat) membranes for cisplatin delivery

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Lung cancer is one of the most common cancer types worldwide, with high mortality rates.However, conventional therapy for lung carcinoma exhibit some drawbacks includingtoxicity, drug resistance, and serious side effects. Nanoparticles are a promising alternativefor the disease treatment, offering a more precise and less toxic therapy [1]. The nanovesiclesshown here were formed by hybrid cell membranes from lung tumor cells (A549) andlymphoblasts (Jurkat) to encapsulate cisplatin, the gold standard chemotherapeutics for lungcancer. First, both cells were collected, washed with a phosphate buffer (pH 7.4) andthen subjected to a hypotonic solution to swell. After centrifugation, the supernatant wasremoved and the pellet redispersed in a lysis buffer to extract the cell membranes. These cellswere homogenized using animal tissue homogenizer. The membranes were obtained by ultracentrifugation at 100,000 gfor 2 hours at 4°C. The pellet containing the plasma membranes was redispersed in PBS with Complete Protease Inhibitor and kept at -80°C until use. A549 cells and lymphoblastsmembranes were coextruded in a 1:1 ratio, at least 30 cycles, through a 200 nmpolycarbonate membrane to form the hybrid nanovesicle. The nanovesicles containing (mHib_Cis) or not (mHib) cisplatin were characterized by dynamic light scattering (DLS)and nanoparticle tracking analysis (NTA). The mHib nanovesicles exhibited a size of 218 nm,PDI of 0.019, zeta potential of -15 mV and concentration of 5.49 x10^8particles/mL. The sizeof mHib_Cis was 207 nm, PDI of 0.112, zeta potential of -15 mV and concentration of 4.51x10^8particles/mL. The biomimetic nanovesicles produced here may be applied in oncologictherapies improving the quality of patients' life.

Acknowledgments:
The funding agency: CNPq 180768/2023-9

References:
[1] - Leger P, Limper A H, Maldonado F. Pulmonary toxicities from conventionalchemotherapy. Clinics in Chest Medicine. 2017; 38(2); 209-222.

Biossensor Based on Gold Nanostars CTAB-Free for Biomedicals Applications

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With the permanence of the COVID-19 pandemic and its rapid rate of contamination, as well as the high capacity for mutations of the SARS-CoV-2 virus, research is still needed to produce efficient, rapid and low-cost tests. Gold nanoparticles (AuNP) are widely reported in the literature as a material that has low toxicity in biological systems and its biocompatibility makes it a promising alternative in the development of nanoimmunoassays for the detection of SARS-CoV-2. There are already approaches in the literature for applications of nanospherical gold bioconjugates with anti-S. Therefore, this work aims to use surfactant-free Gold Nanostars (GNS) in the development of a biosensor for the diagnosis of SARS-CoV-2. The GNS particles have a spherical center and several tips in its morphology, usually elongated, which can confer a larger surface area, thus making it possible to increase the space for bioconjugation reaction and antibody immobilization. Previous works show that the use of surfactants in the synthesis of these particles impairs the bioconjugation, which could prevent a better performance of the biosensor. CTAB-Free gold nanostars (GNS) were synthesized by the seed-mediated citrate reduction method and characterized by UV-vis spectrophotometry, zeta potential, dynamic light scattering (DLS) and transmission electron microscopy (TEM). This work also aims to study the influence of the morphology of nanostars on the immobilization of antibodies and the interaction with the SARS-CoV-2 Spike antigen, comparing it with the AuNP already reported in the literature.

Acknowledgements: CAPES, CNPq e CETENE.

References:

[1] M. M. Vega, A. B. V. Lughi, S. Marsi, S. Carrato and V. Sergo, Journal of Nanoparticle Research, 16, 2729 (2014)

[2] H. Jans, X. Liu, L. Austin, G. Maes and Q. Huo, Analytical Chemistry, 81, 9425 (2009)

Cell membrane-coated nanoparticles for improved glioblastoma treatment

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Glioblastoma (GBM) is a highly malignant form of glioma, with a high risk of recurrence and mortality. Conventional GBM therapy has limited efficacy due to the invasive and heterogeneous nature of the tumor. Currently, the main active molecule used in GBM treatment is temozolomide (TMZ), whose benefits are limited especially due to the resistance mechanisms1. Synthetic chromene-based molecules have proven their potential for cancer treatment, acting on several molecular targets involved in cancer progression2. Here, we investigated cell membrane-coated poly(lactide-co-glycolide) (PLGA) nanoparticles containing novel chromene-based molecules as an innovative anticancer system for GBM treatment. The developed nanostructures coated with the extracted membrane of the U87 human glioblastoma cell line exhibited a diameter of 182 ± 13 nm, and a zeta potential of -10 ± 0.49 mV. Cytotoxicity assays revealed that the cell membrane coated PLGA nanoparticles are more toxic to tumoral cells (U87, U251, and HCB 151) than the uncoated nanoparticles and the free drug, at the same dosage. Additionally, the developed biomimetic system exhibited lower toxicity for non-tumoral cells (HDFn). Our results indicate that the developed nanoparticles may open a new therapeutic opportunity for GBM treatment.

Acknowledgments: This work was supported by the National Council for Scientific and Technological Development (CNPq), Grant 381475/2022-0

References:
1 doi:10.1016/j.gendis.2016.04.007
2 doi:10.1016/j.ejmech.2016.07.057

Characterization of liposomes loaded with temozolomide in the aqueous or organic phase

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Glioblastoma multiforme is the most aggressive variant of glioma. Temozolomide (TMZ) is a prodrug that at physiological pH undergoes hydrolysis to form the methyldiazonic cation. The biopharmaceutical properties of the drug limit its delivery to the tumor contributing to treatment ineffectiveness. Encapsulating TMZ in liposomes reduces biopharmaceutical inconveniences and increases efficacy by promoting greater delivery to tumor cells by exploiting the colloidal characteristics of nanosystems and the tumor microenvironment. The DPPC:Chol:DSPE-PEGmal2000 liposomes were produced and TMZ was encapsulated inthe aqueous or organic phase to compare its properties. The characteristics of mean hydrodynamic diameter (nm), polydispersity index (PDI), zeta potential (mV), and encapsulation efficiency (EE) were evaluated. The liposomes presented as a monodisperse population (PDI 0.2) with small size (136.8±0.07; 120.3±0.029 nm) and negative zeta potential (-22.1; -22.5 mV) for the formulations with TMZ in the aqueous and organic phase,respectively. While the encapsulation efficiency of TMZ was 84.37 and 46.65%, respectively.A PDI of less than 0.2 highlights the efficiency of the extrusion method in control. The smallsize promotes delivery to tumor cells, while the negative charge enhances interaction withbiological membranes in their resting state. The colloidal properties do not change according to the incorporation stage of the drug, but there is a difference in EE. These preliminary results demonstrate that TMZ with its amphiphilic character may beincorporated into any phase of the liposomal system without damaging the colloidal properties, however, the difference in encapsulation efficiency should be considered. Sincethe amount of the drug will be essential to obtain the best biological activity with the lowestpossible dose.

Acknowledgments: CNPq (140779/2021-3) and Fapesp (2021/02609-9)

Cisplatin quantification on PLGA nanoparticles coated with hybrid cell membranes

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Nanotechnology has provided a better therapeutic effect with the nanoparticles- mediated delivery by reducing the toxicity of the drug [1]. Cisplatin is a widely used chemotherapeutic agent that exhibits dose-dependent toxicities in cancer treatment [2]. In this study, we quantify the encapsulated cisplatin in poly(lactic-co-glycolic acid)-based nanoparticles coated with hybrid cell membranes derived from lymphoblast and lung tumor cells (A549)(NP@m) for lung cancer treatment. To this end, we developed and validated a quantificationmethod by ultraviolet-visible spectroscopy for cisplatin derivatized with o-phenylenediamine(OPDA complex; CIS*) at 705 nm following the International Conference on Harmonization(ICH). The nanoparticles were characterized by dynamic light scattering (DLS), nanoparticletracking analysis (NTA), and scanning electron microscopy (MEV). According to the results,this method was selective, accurate (98%), reproducible, and showed repeatability. The limit of quantification is 0,28 µg/ mL of CIS*. The method was also suitable to detect and quantifyCIS* contained in NP@m. The nanoparticles are round with nanometers diameter (142 ±6nm) and negative superficial charge (-6 ±1 mV). The synthesis has 50% of cisplatin encapsulation efficiency and the nanosystems demonstrated effective control of release rates for up to 24 hours. This validation method is sensitive for CIS* quantification, and thenanocarrier shows promise for future in vitro and in vivo experiments.

Acknowledgments: The funding agency: to Fapesp 2022/01434-3.

References:[1] M. Janczura, et al., The Development of Innovative Dosage Forms of the Fixed-Dose Combination of Active Pharmaceutical Ingredients, Pharmaceutics. 14 (2022). doi:10.3390/pharmaceutics14040834.[2] A.N. Shaik, et al., Development and validation of a LC-MS/MS assay for quantification of cisplatin in rat plasma and urine, J. Chromatogr. B. 1046 (2017) 243-249. doi: https://doi.org/10.1016/j.jchromb.2016.11.027.

CO_2 reduction reaction during pulsed laser ablation of gold in water for the control of the regenerative properties of carbon monoxide rich gold nanoparticlesin endothelial tissues

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In the present research we investigate, including negative control experiments with inert gases at the liquid interface, the possibility to induce the CO_2 reduction reaction (CO_2RR) by PLAL of a gold target in water, with the production of carbon monoxide (CO) rich gold nanoparticles (COR-AuNPs). We verify that the CO_2RR controls the biological activity of the produced nanomaterial in endothelial colony forming cells (ECFCs). The biomolecular signaling indicates the ability to behave as carbon monoxide releasing gold nanoparticles, inducing a strong pro-angiogenic activity in endothelial colony forming cells (ECFCs) [1], demonstrated by both *in vitro* and *in vivo* experiments.

Acknowledgments:
This work was supported by CAPES.

References:
[1] M. Li, D. Gallo, E. Csizmadia, L. E. Otterbein, and B. Wegiel. Carbon monoxide induces chromatin remodelling to facilitate endothelial cell migration. Thromb. Haemost, pp. 951-959, 2014.

Comparison of two gold nanoparticle functionalization approaches for SARS-CoV-2 antigen capture

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Gold nanoparticles (AuNPs) have wide properties and can be used in different areas, including biological applications due to characteristics such as biocompatibility, easy conjugation with biomolecules and unique optical properties. AuNPs applications are most of the time related to their shape and [1]. The use of AuNPs for diagnostic purposes is established by the surface chemistry of AuNPs that can be adapted by incorporating different functional moieties, thus, it is possible to modulate the affinity of AuNPs for a wide range of analytes. Among the main methods for surface functionalization of AuNPs are those based on adsorption, covalent binding and affinity [2]. Gold nanoparticles synthed via magnetron sputtering were functionalized by both methodology based on physical adsorption and covalent binding with anti-SARS-CoV-2 antibodies. The colloidal stability of AuNPs was monitored for their characteristics and the ability of antibody-AuNP bioconjugates to recognize their antigen. To assess the relationship with the capture of specific antibodies, an ECLIA-type immunoenzymatic assay was performed, which proved to be efficient in discriminating negative and positive samples for recombinant anti-SARS-CoV-2 protein. The best strategy for capture was given by adsorption functionalization. The RLU values for 1ug/ml of recombinant protein for functionalization by adsorption reached 2.00 x108 compared to covalent binding with lower RLU of 8.00 x107. Limits of detection using ECLIA for both methods revealed detectable values up to 100ng/ml. The ECLIA technique proved to be efficient for capturing the recombinant SARS-CoV-2 nucleocapsid protein. Future studies will be carried out with clinical samples from the nasopharynx to determine the technique's detection limit.

References

[1]. Küünal, Siim et al. Emerging Applications of Nanoparticles and Architecture Nanostructures, p. 411-446 (2018).
[2]. Cordeiro, Mílton et al. Diagnostics, v. 6, p. 43 (2016).

Cytotoxic evaluation of electrospun nanofibers basedon polycaprolactone, cellulose nanocrystals, and *Melaleuca alternifolia* essential oil for tissue regeneration

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Tissue engineering is an interdisciplinary field of scientific research that aims to create functional tissues and organs by the synergistic combination between engineering and biological principles. The functionality of the developed tissue is evaluated according to its ability to efficiently perform, equally to or greater, than the original tissue [1]. In addition, it is of great importance that the developed tissue is biocompatible and does not cause any harm to the recipient organism [2]. Therefore, analyzing the cytotoxicity of the developed tissue is essential for the success of the application. The cytotoxicity assay is a primary requirement of all major standards for biological evaluation of developed tissue. The present study aimed to produce non-cytotoxic nanofibers for tissue regeneration. The nanofibers were prepared by electrospinning technique, using polycaprolactone, cellulose nanocrystals, and *Melaleuca alternifolia*essential oil. The cytotoxicity of the electrospun nanofibers to fibroblasts and keratinocytes was evaluated by indirect MTT assay. Additionally, nanofiber samples were characterized by Fourier transform infrared spectroscopy, scanning electron microscopy, and goniometry. The main results indicated that polycaprolactone nanofibers loaded with cellulose nanocrystals and *Melaleuca alternifolia*essential oil were successfully produced and did not present cytotoxicity. Furthermore, the nanofiber samples became more hydrophilic after the addition of cellulose nanocrystals, which points to a good biocompatibility of the developed tissue.

Acknowledgements:The authors would like to thank to Federal University of Sao Paulo (UNIFESP) and São Paulo State University (UNESP). This research was funded by Fundaçãode Amparo à Pesquisa do Estado de São Paulo (FAPESP), grant number 2019/05856-7; 2020/12507-6; 2021/09335-1.

References:
[1] Pien, N. et al. 2023. Appl. Mater. Today. V. 31, 101737.
[2] Wang, M. et al. 2022. Mater. Today Bio. V. 17, 100495.

Cytotoxic evaluation of gold nanoparticles synthed via magnetron sputtering in mammalian fibroblasts (MRC-5).

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Gold nanoparticles (AuNPs) have been studied in recent decades because they have unique physical, optical and chemical properties such as high surface area, easy conjugation with antibodies and drugs, photothermal characteristics, and its different shapes and s [1]. The search for ecologically sustainable nanostructured materials and their biocompatibility in vitro/in vivo are important characteristics for development and their different applications [2]. Cytotoxicity of gold nanoparticles synthed via magnetron sputtering (MSAuNPs) was analyzed by MTT assay (3-4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) in human fibroblasts (MRC-5) up to a concentration of 50 µg/mL. The preliminary results obtained showed that the nanoparticles presented spherical morphology with varied s. Cell viability tests indicated cytotoxicity in the MRC-5 cell line above a concentration of 35 µg/ml. The production of AuNPs via magnetron sputtering is an eco-sustainable methodology, causing less harm to the environment and more biocompatibility to tissues. Further studies are in focus in order to understand the in vitro mechanisms of action of MSAuNPs with promising applications in the health and environment areas.

[1] M. Nejabat et al., 2023; Journal of controlled Release, vol.354, 221-242.
[2] P.B. Santhosh, J. Genova, H. Chamati, 2022; Chemistry, vol. 4 (2), 345-369.

Cytotoxic potential of gold nanoparticles synthed by the green route with the extract of the Antarctic macroalgae *Palmaria decipiens* in mammalian fibroblasts

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Gold nanoparticles (AuNPs) stand out for their advantages such as biocompatibility, adjustable optical properties and easily changeable surface chemistry, being widely used as carriers of drugs and molecules to improve the diagnosis and treatment of diseases (BOTTEON, 2021). This work reports the biocompatibility of gold nanoparticles synthed by green route based on extracts of the Antarctic macroalgae *Palmaria decipiens*(PCAuNPs). The use of biological approaches in the development of ecologically correct processes for the production of PCAuNPs reduces the environmental impact and often reduces the cytotoxicity of these materials against mammalian cells. Antarctic macroalgae produce substances with important biological activities such as antiviral, anti-inflammatory and antimicrobial actions, among others (GIUSTINA et al, 2022). The cytotoxicity of gold nanoparticles biosynthed with the methanolic extract of the Antarctic macroalgae *Palmaria decipiens*was analyzed using the MTT assay in mammalian fibroblasts (L929). Preliminary results obtained showed that PCAuNPs presented spherical morphology with varying between 40 and 60nm. Cell viability tests indicated absence of cytotoxicity in the L929 strain at concentrations up to 100 μg/ml, with an IC50 of 190.27 μg/ml. The characterizationof PCAuNPs synthed by the green route, as well as their antimicrobial and antifungal potential are the next steps of this research to better understand their applicability.

Designing bioactive nanocarriers for the delivery of neurogenic and neuroprotective drugs

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Mesoporous bioactive glass (MBG) has a unique porous structure that makes it an ideal candidate for use as a drug nanocarrier and delivery system [1]. This is especially interesting for drugs that have low aqueous solubility, fast degradation, low oral bioavailability, and inadequate tissue permeability [1,2]. MBG has excellent solubility in water, good stability, low toxicity, high cellular uptake, remarkable biocompatibility [1,2] and considerable tissue permeability, especially through the blood-brain barrier [2]. The aimof this study is to synthesize mesoporous bioactive glass nanospheres derived from the SiO2-CaO-ZnO-CuO-B2O3 system (in mol%) obtained by the sol-gel method with combined dual functions of bioactivity and controlled drug delivery, and to further investigate their delivery property of neuroprotective drugs as well as the functional effect on neural cellsthrough bioinorganic ion therapy. The morphologies, microstructure, specific surface areasand pore sizes of MBGs were characterized by microscopy, vibrational spectroscopy andnitrogen physisorption, respectively. MBG nanospheres with spherical morphology and internal mesoporous microstructures were successfully prepared by a sol-gel method, exhibiting high specific surface area and mesopore volume. The MBG nanospheres demonstrate excellent bioactivity by inducing apatite mineralization in simulated body fluidsand effective ionic release into this microenvironment. In addition, it to distinct degradability, high charging efficiency and neuroprotective functions. These unique characteristics suggest that the obtained MBG nanospheres may be used for the neural tissue engineering. Acknowledgements:This work has been supported by the following Brazilian research agencies: CAPES (88887.711128/2022-00) and FAPESP (2022/03247-6).References:[1] K. Zheng, W. Niu, B. Lei, A.R. Boccaccini, Acta Biomaterialiavol. 133, 168-186 (2021).[2] W. Jong, P. Borm, Int. J. Nanomedicine vol: 3(2), 133-149 (2008).

Design of Experiments Approach for Formulation of PLGA Nanocarrier System Loaded with the Anticancer Agent QPhNO2 for Efficient Drug Delivery Applications.

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Cancer is a global public health issue causing numerous deaths. Traditional treatments can cause severe side effects. Nanotechnology offers a promising alternative for cancer treatment, using nanoparticles to deliver drugs directly to the disease site, reducing side effects and improving efficacy. $QPhNO_2$is a naturally-derived molecule with potential anticancer propertie however, its low water solubility hinders clinical development. Nanoencapsulation with PLGA nanoparticles aims to increase solubility, protect the molecule, and improve efficacy.

This study encapsulates $QPhNO_2$in PLGA nanoparticles for anticancer drug use. Stability and drug release kinetics will be investigated. Nanoencapsulation improves solubility of low-water-solubility anticancer molecules.Materials and methods include PLGA, ethyl acetate, Poloxamer 188, and trehalose. A 2^3factorial design optimizes PLGA nanoparticle formulations, analyzing zeta potential, , and PDI. Nanoparticles were prepared using the Emulsification Solvent Diffusion method. Release kinetics, encapsulation efficiency, and in vitro cytotoxicity assays are ongoing. In conclusion, this study develops PLGA nanoparticles containing QPhNO2 for enhanced cancer treatment efficacy and reduced side effects. Further pre-clinical research is needed to evaluate its safety and efficacy as a cancer treatment.

Acknowledgements:

LIMAV, UFPI, CNPQ e CAPES.

References:

[1] Misra R, Acharya S and Sahoo SK. Cancer nanotechnology: application of nanotechnology in cancer therapy. Drug Discov Today; 842-50(2010).

[2] Sadat Tabatabaei Mirakabad and Nejati-Koshki K, et al. PLGA-based nanoparticles as cancer drug delivery systems. Asian Pac J Cancer Prev; 15(2):517-35 (2014).

Development and characterization of doxorubicin and quinizarin nanoemulsions for application in Photodynamic Therapy and Chemotherapy of oral squamous cell carcinoma

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Due to their high complexity and resistance, oral squamous cell carcinomas (OSCC) have a difficult therapeutic process and often translate into sequelae or a range of harmful side effects. Thys nanoemulsions (NE) are capable of providing an efficient alternative for the treatment of advanced oral cancer, based in Photodynamic Therapy (PDT) technique and Chemotherapy from delivery of quinizarin (QNZ - photosensitizer) and doxorubicin (DOX - antineoplastic agent), both are responsible to interact with DNA and can modulate several cytotoxic effects through the photodynamic processes induced in tumor cells. NEs system was prepared by the interfacial deposition method with Pluronic F-68 surfactant, soybean phosphatidylcholine, with concentration of 0.05 mg/mL of QNZ in the presence and absence of the DOX at concentration of 1% w/v. For characterization of particle size, polydispersity index and zeta potential were measured by DLS technique. Analysis in absorption and fluorescence emission spectroscopy in 2D/3D mode were performed and used for photophysical characterization as well as to test the quantification and determination of the incorporation efficiency of QNZ and DOX in the formulation. For the unloaded NE and NE/QNZ, NE/DOX and NE/QNZ/DOX, the particle size were 228.1 ± 65.7nm, 200.9 ± 34.5nm, 208.8 ± 46.9nm and 180.8 ± 8.3nm respectively. NEs presented themselves in nanometer scale and monodisperse, with adequate zeta potential between −22mV and −31mV. Through the spectrofluorimetric method developed with a value of $R^2 > 0.99$ we found a high incorporation efficiency of QNZ and DOX in NEs, that was 86% and 95% respectively. Therefore, the nanomaterials were considered promising for synergistic application in antitumor PDT. This work is supported by FAPESP, nº 2022/11326-6 (Grants R.B.C.) and CNPq Universal Project nº 404416/2021-7 (Grants F.L.P).

Development and validation of a method for simultaneous quantification of Erlotinib and Doxorubicin in cell membrane-derived nanovesicles

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The use of cell membrane-derived nanovesicles as drug delivery systems has emerged as a promising strategy to improve the therapeutic efficacy of drugs and as well as to overcome difficulties associated with conventional cancer treatments such as chemoresistance and high toxicity. However, quantifying drug loading efficiency in these systems is crucial for their successful application. Validating the quantification method guarantees the reliability and reproducibility of the data. In the present study we developed a new method for the simultaneous quantification of erlotinib and doxorubicin via high-performance liquid chromatography (HPLC). The method was validated following the International Council for Harmonization (ICH) guidelines, which establishes criteria for accuracy, precision, specificity, and robustness. We applied this validated method to determine the drug loading efficiency of these two drugs in membrane nanovesicles derived from A549 cancer cell lineage. The validation of our method showed that it is linear (erlotinib R2= 0.999; doxorubicin R2 = 0.999), accurate and precise, with a limit of detection (LD) and limit of quantification (LQ) of 0.18 and 0.56 μg/mL for erlotinib, and 0.1 and 0.29 μg/mL for doxorubicin. We also showed the method is specific and robust, and that it can accurately quantify both drugs in the presence of potential interferences. The drug loading efficiency of erlotinib and doxorubicin in the membrane nanovesicles was approximately 50% and 30%, respectively. These results demonstrate the potential of this novel validated method to accurately and simultaneously quantify the drug loading efficiency of erlotinib and doxorubicin in nanovesicles. Moreover, this study contributes to the development of new and safe drug delivery systems for cancer therapy.

Development of a multifunctional magnetic-fluorescent nanomaterial (Fe_3O_4@$mSiO_2$-Cur) and evaluation of its potential for the treatment of topical diseases by photodynamic therapy combined with magnetic hyperthermia

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This work aimed to develop a magnetic-fluorescent nanosystem composed by magnetic nanoparticles (MNP) covered by mesoporous silica and containing curcumin as photosensitizer adsorbed on its surface (Fe_3O_4@$mSiO_2$-cur). The purpose of the system is the application in photodynamic therapy combined with magnetic hyperthermia, in order to treat, topically, skin diseases that cause difficult-to-heal wounds, such as the neglected diseases sporotrichosis and leishmaniasis. The use of curcumin in photodynamic therapy is based on its photosensitizing properties, which make it capable of generating reactive oxygen species when activated by light that are harmful to diseased cells, such as cancer cells, and microorganisms, such as fungi, for example. The magnetic nanoparticles were synthed by the coprecipitation method and encapsulated in silica using CTAB as a pore forming agent. The resulting material has an average particle of 12±3 nm, with a silica layer thickness of 3 nm and pore of 4 nm, results obtained by transmission electron microscopy and N_2adsorption isotherm (BET). The adsorption of curcumin on the porous silica matrix was confirmed by EDS and fluorescence optical microscopy. The ILP (Intrinsic loss power) values resulting from the magnetic hyperthermia analysis were 0.63 and 0.35 $nH.m^2.Kg^{-1}$forthe $Fe_3O_4$and Fe_3O_4@$mSiO_2$-cur NPMs, values consistent with the literature and confirmingthe potential treatment by magnetic hyperthermia. In vitro cell internalization assays were performed on sporothrix brasiliensis and Leishmania amazonensis, and a satisfactory internalization profile of the magnetic-fluorescent nanosystem was observed. The results obtained demonstrate that this nanosystem has characteristics suitable for the application of combined treatment of topical diseases, such as sporotrichosis and leishmaniasis, by photodynamic therapy combined with magnetic hyperthermia. Acknowledgements: The authors would like to thank CNPq and FAPERJ.

Development of a nanocomposite based on usnic acidand conjugated polymer for use as bioherbicide

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The growth and demand for food production have been accompanied by an increase in the use of pesticides such as herbicides, insecticides, and fungicides [1]. However, the indiscriminate use of these pesticides has been associated with environmental risks and effects on human health [1]. Therefore, many efforts have been directed towards identifying interactions between contaminants and ecosystem structure to prevent unintentional contamination of these molecules in the environment [2]. In this scenario, the use of nanotechnology presents itself as an alternative to mitigate the risks that these agricultural chemicals cause to the environment. Among the different existing release systems, conjugated polymer nanoparticles (NPCs) emerge as very promising systems [3]. Usnic acid (UA) is a new bioherbicide with potential directed towards the inhibition of photosystem II (PSII), and new formulations derived from nanostructured materials may be a potential improvement for its herbicidal activity. In this study, nanoparticles of the conjugated polymer poly(p-phenylenevinylene) (CNPPV) were loaded with UA to form a new nanocomposite (NPCs-UA) to explore its application as a bioherbicide. The results showed that the nanoformulation had a diameter of 162.1 ± 75 nm, polydispersity of 0.245, and zeta potential of -20.05 ± 4.48 mV. The data also revealed a different release profile of NPCs-AU when compared to free UA. The results showed a slower release for NPCs-UA and an inhibitory effect on the photosystem II of the model plant Brassica juncea.

Acknowledgements:
This research was funded by, CNPq and, SISFÓTON-MCTI, , FUNDECT and for financial support. - Brazil, CAPES.

References:
[1] ABELHA, T. F. et al. Journal of Materials Chemistry B, jan. 2020.
[2] BROGAN, W. R.; RELYEA, R. A. Multiple mitigation mechanisms, jan. 2017.
[3] OLIVEIRA, H. C. et al. Nanoencapsulation Enhances the Post-Emergence Herbicidal Activity of Atrazine against Mustard Plants, 17 jul. 2015

Development of bevacizumab encapsulated PLGA nanoparticles for lung carcinoma therapy

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Lung cancer is a malignant neoplasm of high incidence and mortality, occupying the second place among the most common in the world (International Agency for Research on Cancer - IARC). Although great advances in treatment have been achieved, several drugs still have limitations regarding stability and delivery, making it necessary to search for alternatives to increase their efficiency. This project aims to develop a delivery system based on PLGA nanoparticles for encapsulation and delivery of bevacizumab (BVZ), a antibody that inhibits pro-angiogenic factors in the treatment of lung cancer (NSCLC). PLGA nanoparticles were obtained through the double emulsion method. The PLGA-BVZ were characterized in their hydrodynamic diameter, polydispersity index (PDI) and zeta potential. The sizes were between 171 and 237 nm, PDI between 0.14 and 0.22, the zeta potential between -23 and -27 mV. In addition, 14 days stability tests and Fourier-transform infrared spectroscopy (FTIR) were performed. The results showed that the formulations were stable during the analyzed period. The FTIR results pointed to specific non-covalent interactions occurring between PLGA and BVZ. The results are promising and may contribute to future treatments against lung cancer.

DEVELOPMENT OF LIPID NANOPARTICLES FOR CHEMOTHERAPEUTICS DELIVERY

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Solid lipid nanoparticles (SLN) represent a class of important nanocarriers sincethey can encapsulate lipophilic drugs, which can improve their solubility in waterand, consequently, their bioavailability. Photodynamic therapy (PDT) is alsoconsidered a treatment modality for different types of neoplasms. This therapyconsists of the combination of light, photosensitizer (PS), and molecular oxygen(O2), capable of generating oxidative products that can cause cell death.Herein, we developed SLN to encapsulate Paclitaxel (PTX) and the PS 5-aminolevulinic acid (5-ALA) to combine both therapies as an alternative strategyfor lung cancer therapy. The nanoparticles were obtained using a high-energymethod. Compritol ATO 888 was chosen as the lipid matrix, and Poloxamer 188as the surfactant to stabilize the nanoparticle dispersion. Two differentsyntheses were tested, namely SLN_1 and SLN_2, both resulting innanoparticles with average sizes between 169 nm and 170 nm, polydispersityindex (PDI) around 0.3, and negative zeta potential (-26 mV and -28 mV).Stability tests were carried out for 28 days and the results indicated that theformulations remained stable during storage at 4°C, being promising deliverysystems to further studies of lung cancer therapy.

Development of polymeric nanoparticles containing bevacizumab as a new technological platform for targeted drug delivery for the treatment of colorectalcancer

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Colorectal cancer (CRC) is a public health problem; among the types of cancer, it ranks third in incidence and second in mortality [1]. The conventional therapeutic resource for the treatment of cancer is intravenous therapy. However, the lack of selectivity/specificity of antineoplastic agents and their distribution to healthy tissues/organs requires administration in high doses, which can result in toxicity. Among the antineoplastic agents used to treat CRC, bevacizumab (BVZ) stands out as a first-line drug. The encapsulation of BVZ, an angiogenesis-inhibiting antibody, in nanocarriers, has been explored to increase its efficacy in treating colorectal cancer (CRC). The present project aims to develop nanoparticles (NPs) of GG and BVZ by the polyelectrolytic complexation method and modify their surface with chitosan (CS). Different proportions of polymers were tested to obtain the NPs, showing average diameters in the range of 222.6 to 412.1 nm, zeta potential from -16.3 to -40 mV, and PdI from ~222 to 412. Chitosan promoted charge inversion (>+40 mV) in addition to increasing the particle diameter (268 to 400 nm) and the encapsulation efficiency, ranging from 29 % to approximately 60 %. NPs exhibited good physical stability and good mucoadhesive properties by interaction analyses on DLS, NTA, and rheology. The biocompatibility of NPs was evaluated in HCT116, HPMEC cells, human fibroblasts, and macrophages in 2D models and 3D spheroid models, exhibiting dose- and time-dependent cytotoxicity. In addition, the antiangiogenic activity of the nanoparticles was evaluated by the chicken egg chorioallantoic membrane (CAM) model, exhibiting good antiangiogenic properties.

Acknowledgements:

The authors are thankful to FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo) Grant 2019/10761-5 and Grant 2022/14075-1.

References:

[1] International Agency for Research on Cancer (IARC)., The Global Cancer Observatory, (2020).

DEVELOPMENT OF RADIOACTIVE NANOPARTICLES FUNCTIONALIZED WITH GUM ARABIC TO BE USED IN NANOBRACHYTHERAPY

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The development of new materials emerges as an alternative to the treatment of cancer, from the combination of nanotechnology and brachytherapy a new area of research was born, Nanobrachytherapy, which through the properties of nanometric materials can achieve better results in the fight against cancer. The objective of this work is classified as radiotherapy, which consists of the use of ionizing radiation to destroy or inhibit the growth of abnormal cells that form a tumor. [1] The ability to integrate NPsAu into biological systems is due to the nanometric dimensions of NPsAu probes which facilitate their incorporation into biological systems, as well as their bioconjugation and non-cytotoxic potential. [2] Taking into account the previous objective, gold was selected as the base element for obtaining nanometric systems, which due to its chemical richness and especially due to the intrinsic properties of one of its radioisotopes, which would allow us in theory to meet the stated objective. . Initially, it was based on the knowledge of the literature, and non-radioactive nanometric systems were obtained, and after a series of stability, characterization and application tests, the radioactive nanometric systems were obtained. Working with radioactive systems posed a great challenge, and up to now it presents us with situations to solve, but we have managed to create a methodology for obtaining, characterizing and applying radioactive gold nanoparticles, and also obtaining positive results from their application.

CNPq, Fapesp 2017/50332-0, IPEN 2018.05.IPEN.09, IAEA BRA-6026

References:

[1] E. Evans and J. Staffurth, “Principles of cancer treatment by radiotherapy,” Cancer Treat. Rev., vol. 36, no. 3, pp. 111-116, 2017, doi: 10.1016/j.mpsur.2017.12.006.

[2] I. H. El-sayed, X. Huang, and M. A. El-sayed, “Surface Plasmon Resonance Scattering and Absorption of anti-EGFR Antibody Conjugated Gold Nanoparticles in Cancer Diagnostics : Applications

Direct synthesis of polymer-stabilized gold nanoparticles mediated by nitrogen-containingpolymers

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The unique properties of gold nanoparticles (AuNPs) enable their use in a wide array of applications. The straightforward applicability of AuNPs requires the development of facile synthetic approaches.[1] Commonly employed AuNPs synthesis involve the reduction of gold ions using reducing agents in the presence of stabilizing agents, in a multi-step process. In this context, the use of nitrogen-containing polymers allows for a fast one-pot synthesis of AuNPs where the polymer chains act as both reducing and stabilizing agent.[2] In this work, we used a library of nitrogen-containing polymers to produce polymer-stabilized AuNPs: i) polyethylenimine and derivatives, containing primary to tertiary amino grou ii) poly(aminoethyl)methacrylates, containing only tertiary amino grou and iii) polyoxazolines, containing amide groups. AuNPs synthesis is only feasible at pH >5, thus the protonation degree of the chains is an important parameter. The experimental data suggest hydrodynamic diameters (dH) ranging from 10 to 150 nm. However, since DLS is heavily weighted by particle volume, most of the produced AuNPs are likely to have a dH within 10-30 nm. This is corroborated by UV-vis spectroscopy, where the maximum absorption (λmax) obtained was in 520 to 545 nm range. Surface ζ-potential values in the range -15 to +15 mV were determined, yet the AuNPs were reasonably stable in PB 10 mM pH 7.4, suggesting a synergic electrostatic and steric stabilization. These preliminary findings are expected to potentialize advances towards the practical and ready-to-use manufacturing of polymer-stabilized AuNPs and other metal nanoparticles with applications in a myriad of different fields.

Acknowledgements: We acknowledge the sponsoring by FAPESP (grants 2019/06634-8 and 2021/12071-6) and GBM the fellowship by FAPESP (grant 2022/13395-2).

References:
[1] E.C. Dreaden, et al. Chem. Soc. Rev. 41, 2740-2779 (2012).
[2] C.A.S. Ribeiro, et al. J. Colloid Interface Sci. 614, 489-501 (2022)

DNA-Templated Self-Assembly of the HypotensivePeptide Bradykinin

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Bradykinin (BK) is a short peptide hormone with a vasodilator effect, belonging to the kininase-kallikrein system, and related to the release of inflammatory mediators. BK has been explored as a blood pressure reducer, and is a well-established active ingredient for antihypertensive drugs in the pharmaceutical industry. However, there are no reports on nanostructured BK materials. Des-Arg9-BK (DBK) is an important BK analog, with a sequence almost identical to that of BK but without an arginine residue. DBK has a different mechanism of action, being a B1 receptor agonist while BK is a B2 agonist. Our objectives were to produce supramolecular structures based on BK and DBK, provide detailed structural data on these assemblies, and investigate whether they exert cellular responses like those of the peptide in its monomeric form. BK and DBK were complexed with DNA fragments. Fluorescence spectroscopy determined the critical aggregation concentrations, while circular dichroism experiments analyzed the secondary structure. Atomic force microscopy and small-angle X-ray scattering investigated the nanoscopic structure of the complexes. Calcium influx analysis in HuVEC cells evaluated the cellular response. We discovered that DNA acts as a template for BK ribbons, leading to the formation of nanoscopic fibrils stabilized by β-sheets. Interestingly, this effect is not observed for DBK. Furthermore, BK preferentially binds to the major grooves of DNA duplexes, while DBK intercalates between nucleotide bases. Importantly, BK-DNA fibrils were found to modulate calcium influx in HuVEC cells, thus preserving the bioactivity of the native peptide. We demonstrate a feasible strategy to fabricate BK-based nanostructured matrices that are potentially exploitable for the development of biomaterials endowed with hypotensive capabilities. for the development of biomaterials endowed with hypotensive capabilities.

Acknowledgements: To FAPESP (grant n° #2022/03056-6) and CAPES.

Effects of Bisphenol-A in giant unilamellar vesicles as mimetic models of a tumoral cell membrane.

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One of the greatest challenges facing humanity relates to the availability and quality of water. Every day several chemical compounds are dumped directly or indirectly into nature and end up entering human and animal consumption. One of the most worrying classes is the emerging pollutants that cause interference in the endocrine system. One is Bisphenol A (BPA) [1], a compound used in manufacturing plastics, present in packaging and resins, and released by hydrolysis. Another concern is about the effects of this compound on tumoral cells. Thus, this work aims to study the effects of the pollutants on tumoral cell membrane models from the creation of giant unilamellar vesicles (GUVs) [2], which mimic biological membranes. The lipids used in the construction of GUVs were DOPC and DPPS, together, ata ratio of 80/20, or separately, to analyze the effects of BPA at a concentration of 1x10-4 mol/L. Through phase contrast microscopy, it was observed that after contact with the pollutant, the vesicles became unstable, losing their spherical shape, and increased in ; adjacent formations appeared inside (endocytosis of the external environment) and outside (buddings). In conclusion, the presence of Bisphenol A in the external environment induced diverse effects in the bilayers of DOPC and DPPS, and additional studies must be carried out to unravel the specific molecular interaction responsible for the effects.

Acknowledgments: CNPq, SPBMAT, FAPESP, and CAPES.

[1] Tarafdar A, Sirohi R, Balakumaran PA, et al. The hazardous threat of Bisphenol A: Toxicity, detection and remediation. J Hazard Mater. 2022;423(PA):127097. doi:10.1016/j.jhazmat.2021.127097.

[2] Angelova, M. I., Dimitrov, D. S., Faraday Discussions, 1986, 81, 303-311.

Encapsulation of antimicrobials in lipid corenanocapsules

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Substantial advances happened in the development of functional nanomaterials towards biomedical materials, mainly for targeted therapies and drug delivery. Lipid core nanocapsules (LCN), a class of nanocapsules in which a polymeric shell is arranged around an inner core and is formed by a dispersion of a liquid lipid and a solid lipid, have gained attention since they can vehicle actives soluble in their lipidic core or polymeric shell [1]. Our objective was to encapsulate fluconazole and rifamycin SV in LCN, aiming to improve existing treatments for fungal and bacterial infections by controlling their release. Empty-LCN (unloaded), Flu-LCN (LCN loaded with fluconazole) and Rif-LCN (LCN loaded with rifamycin SV) were produced with mean diameter around between 220 and 230 nm, PDI of 0.13 to 0.15, and ζ-potential of -27mV. Fluconazole presented a type II distribution in LCN, with encapsulation efficiency of 8.0%. On the other hand, rifamycin SV presented a type III distribution, with encapsulation efficiency of 66.2%. Around 53% of fluconazole was released in the first 7h, reaching 77% in 48h; while less than 5% of rifamycin SV was released in the first 8h, reaching 21% in 48h. Empty-LCN did not show any in vitro antimicrobial activity, while loaded-LCN showed similar MIC to free antimicrobials when tested S. aureus, P. aeruginosa and C. albicans microorganisms [2]. Thus, LCN can be considered a promising drug delivery system for antimicrobials, which could be further evaluated for topical applications.

Acknowledgements: FAPESP (grants 2018/07707-6, 2018/06475-4, 2020/11333-4, 2022/08563-3)

References:

[1] Venturini, C.G. et al. Formulation of lipid core nanocapsules, Colloids and Surfaces A: Physicochemical and Engineering Aspects, 375(1):200-208, 2011.

[2] Methods for dilution antimicrobial susceptibility tests for bacteria that grow aerobically; approved standard-sixth edition. Wayne, PA: CLSI - Clinical and Laboratory Standards Institute, 2003.

Encapsulation of phenolic-rich extract from jambolanby nanoprecipitation technique

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Jambolan (*Syzygium cumini*L.) has been studied as a source of antioxidants and food colorant due to its phenolic composition, which includes anthocyanins [1]. This study aimed to encapsulate an extract rich in anthocyanins (ANT) and other phenolic compounds (PC) from jambolan obtained by ultrasound-assisted extraction [2]. For encapsulation, two formulations [2.2 g extract (20 mg ANT; 97 mg PC), 0.5 g pectin, 1.5 g whey protein concentrate (C) or isolate (I)], were precipitated in two antisolvent phase compositions: absolute acetone (C100, I100) or 50% v/v (C50, I50). The particles were characterized by PC and ANT encapsulation efficiency (EE%), particle by DLS, Zeta potential, FTIR, and X-ray diffraction. The results for EE% of ANT [C100: 56.89% (1.29); C50: 48.65% (1.74); I100: 35.24% (1.24); I50: 36.33% (2.76)] differed ($p < 0.05$) between formulations, however, EE% of PC [C100: 82.39% (1.59); C50: 73.81% (6.72); I100: 84.61% (1.64); I50: 75.80% (6.91)] did not differ ($p > 0.05$). Zeta potential at pH 4.7 varied from −8.3 to −10.3 mV. Particle s of WPI-based formulations [I100: 661.3 nm (79.2); I50: 302 nm (27.6)] were smaller than WPC-based formulations [C100: 1263.8 nm (124.7); C50: 1156 nm (60.4)]. FTIR showed that all encapsulated presented similar spectra to those of the proteins used, in addition to displacements and formation of new bands (1072 to 1022 cm^{-1}in C100, C50, and I50 and in 1072 and 1026 cm^{-1}in I100). The diffractograms showed that all encapsulated presented an amorphous structure. These results show the potential of nanoprecipitation and the combination of encapsulating agents in producing particles containing jambolan phenolic-rich extract, which can protect and optimize the application of these compounds.

Acknowledgment:

National Council for Scientific and Technological Development.

References:

[1] I.M. Tavares et al. Food Res Int., 82:1-13 (2016)

[2] L.B.S. Sabino et al. Food Bioprod Process.,127:77-89 (2021)

Endolysin PlyB221-decorated Silver Nanoparticles as Promising Nanomaterials for Antibacterial Applications in Agriculture

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The emergence of super-resistant bacteria in livestock is a worldwide problem. Silver nanoparticles (AgNPs) have received significant attention in recent years due to their potential use as antibacterial agents. Once synthesized, AgNPs can interact with biomolecules, including proteins, which can lead to the formation of a protein corona around the particle. Endolysins from bacteriophages have also been highlighted as biomaterials for therapeutic applications. Endolysin PlyB221 has a modular structure composed of enzymatically active domains (EADs); and a cell wall binding domain (CWBD) that recognizes and brings endolysin to cell wall associated ligand molecules with high specificity. In this study, AgNPs was synthesized using a chemical reduction method and characterized using various techniques, including transmission electron microscopy, dynamic light scattering and UV-Vis spectroscopy. The production of the recombinant endolysin PlyB221 was evaluated in SDS-PAGE, and the interaction of AgNPs with PlyB221was evaluated by monitoring the size, zeta potential, and the absorption band in the UV-Vis. Our results showed that AgNPs were successfully synthesized with a mean diameter of 7 nmand a zeta potential of -30 mV, and the PlyB221 with a mean diameter of 144 nm and a zetapotential of -13,5mV. The interaction of AgNPs with endolysin showed a significant increasein size for 2300nm and variation of the zeta potential for -8mV. Through UV-Vis it was possible to verify a shift in the silver absorption band (396 nm to 420nm), whichdemonstrates the interaction with endolysin, leading to the formation of a corona protein. These findings have implications for the development of theranostic platforms for use in agriculture.

Establishment of an in vivo platform to evaluate theeffects of zinc nanocomposites and nanoconjugates with curcumin in an Alzheimer's model

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Alzheimer''s disease (AD) is a fatal neurodegenerative pathology, for which there is no cure. This scenario is intensified by exposure to nanoparticles due to their neurodegenerative effects. Curcumin, a bioactive compound, in addition to present neuroprotective effects, can be used as a biomarker of AD. As some pharmacokinetic properties of this bioactive interfere with its therapeutic effects, the nanoformulation is a tool that can be used as a drug delivery system. Drosophila melanogaster is a well-established model organism that allows the creation of mutant AD-related phenotypes and enables a rapid assessment of the possible effects of nanomaterials. Therefore, our main objective was to establish an in vivo platform to evaluate the effects of nanocomposites and nanoconjugated curcumin in the AD model. Through the UAS-GAL4 system and lineages of flies carrying human mutations in the amyloid precursor protein (APP), we performed the expression of the mutant protein in the central nervous system during development, leading to neurological and motor damage anda decrease in life expectancy. We evaluated the effects of the nanomaterial Simonkolleite,pure and conjugated with curcumin, in our AD model analyzing life expectancy, motor behavior and toxicity in the eyes of affected animals. Our experiments culminated in the establishment of a model in which expression of the APP carrying a human mutation leads to severe neurodegeneration, premature death and cellular toxicity in the animals eyes. Together, we were able to develop a platform for screening biomolecules conjugated to nanomaterials that may contribute to the development of new therapies for AD. We are currently testing whether Simonkolleite conjugated to curcumin can play a neuroprotective role in our in vivo Alzheimer model, reducing the damage caused by exposure of the central nervous system to nanomaterials throughout life and in the establishment of neurodegenerative diseases.

Establishment of an *in vivo* platform to evaluate the effects of nanocomposites and nanoconjugated Curcumin in a C9orf72 Amyotrophic lateral sclerosismodel.

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Amyotrophic lateral sclerosis (ALS) is a fatal and incurable disease characterized by progressive degeneration of motor neurons. This scenario is intensified by the exposure to nanoparticles due to their neurodegenerative effects. Studies demonstrate the effectiveness of Curcumin, a bioactive compound, in neurodegenerative disorders, however, the effects of this natural compound in ALS have not yet been fully elucidated, since some pharmacokinetic properties affect its therapeutic effects. To overcome such limitations, nanoformulation is a tool that can be employed. *Drosophila melanogaster* is a well-established model organism that allows creating mutant phenotypes related to ALS and enables quick evaluation of the possible effects of nanomaterials since it is an economical and dynamic model. Our main goal was to establish an *in vivo* platform to evaluate the effects of nanocomposites and nanoconjugated Curcumin on an ALS model. In order to express the C9orf72 ALS transgene containing 30 hexanucleotide (G4C2) repeats, we used the UAS-GAL4 system with drivers that control the gene expression on the eyes and motor neurons of the animals. The animals completed their development at 29°C to increase the transgene expression. We evaluated the effects of the nanomaterial Simonkolleite, pure and conjugated with Curcumin, on our ALS model based on lifespan, larval and adult motility, and eye cell toxicity in affected animals. Transgene expression caused severe eye toxicity compared to control, while its expression in motor neurons led to severe degeneration, causing a decrease in lifespan. We are evaluating the neurodegenerative potential of the nanocomposite. Therefore, we were able to establish a model that mimics the phenotypic characteristics of ALS patients. Our hypothesis is that Curcumin performs a neuroprotective role in our *in vivo* model, reducing the damage caused by nanomaterials.

Evaluation of antitumor effect of PEGylated nickel ferrite nanoparticle-mediated magnetohyperthermia

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Despite extensive advances in the field of radiotherapy, chemotherapy and surgery, improving survival and quality of life for cancer patients has still been a challenge. For this reason, investigations combining conventional therapies with other modulators are performed [1,2]. In this work, we investigated the effect of magnetohyperthermia (MHT) induced by PEGylated nickel ferrite (PEG-NiF) nanoparticles on breast cancer (MCF-7) cells. The nanoparticles (NPs) were synthetized by hydrothermal method and characterized by X-ray diffractometry, dynamic and electrophoretic light scattering, thermal analysis, infrared spectroscopy, Mössbauer spectroscopy and vibrating sample magnetometry. PEG-NiF NPs heating profile using AC magnetic field of 300 A, with 279 kHz of frequency, was studied at different concentrations (100, 200 and 350 $\mu g.mL^{-1}$) and correlated with their potential cytotoxic effect against MCF-7 cells. The results evidenced the formation of spinel phase with crystallite size of 25.6 ± 0.5 nm, hydrodynamic size below 150 nm, saturation magnetization of 53 $emu.g^{-1}$ and superparamagnetic behavior at room temperature. The heating response was tuned by PEG-NiF NPs concentration reaching 42 ºC at 350 $\mu g.mL^{-1}$. Cell viability remained unaffected by the application of the AC magnetic field alone. On theother hand, when it is combined with increasing concentrations of PEG-NiF NPs the inducedMHT was harmful to MCF-7 cells resulting in up to 83 % of cell death. This antitumor effectagainst MCF-7 cells was 22 % greater than that of PEG-NiF NPs alone. Based on theseresults, we could envision the application of the PEG-NiF NPs as a magnetohyperthermiaagent.

Acknowledgements:

This work was supported by CNEN, CDTN, FAPEMIG, CNPq and CAPES.

References:

[1] SENGEDORJ, A. et al. Cancers, 14(9), (2022).

[2] KANG, J. K. et al. Arch. Pharm. Res., 43, 46-57, (2020).

Evaluation of silver nanoparticles nanofibers with red propolis from Alagoas: characterization and potential antimicrobial application

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The nanofibers were obtained by a solution of silver nanoparticles (NPAg-P) with red propolis from Alagoas (RPA) incorporated into the polymer matrix of polycaprolactone (PCL). The electrospun nanocomposites were named PCL-E, E1%, E2% and E5%. The characterization of the material was performed by: X-Ray Diffraction (PXRD), thermogravimetry (TG), Differential Exploratory Calorimetry (DSC), Scanning Electron Microscopy (SEM), mechanical test, release and antimicrobial activity. Metallic silver crystalline planes visualized by PXRD confirm the obtainment of NPAg-P. By TG, it was possible to observe that the decomposition of the products compared to the pure polymer did not change, but the DSC curves decreased in the melting temperature because of the reduction of the crystallinity of the compounds due to the increase of NPAg-P. The morphology of the nanofibers was evaluated by MEV and it was observed that at E5% there was a decrease in the fiber and greater uniformity of distribution, when compared to the other concentrations. In the mechanical test, it was observed that the addition of silver resulted in a decrease in the elasticity and resistance of the material, compared to pure PCL fiber. For the release test, there was a release of 0.0833 µg/mL in 24h. In the antimicrobial activity, inhibition halos (8.0 mm) were observed for the evaluated bacteria with the E5% nanofibers. For isolated E5%, inhibition halos of, on average, 8.0 mm were observed. For RPA, a halo of 6.5 mm was observed. It was observed that NPAg-RPA nanofibers are a promising antimicrobial agent, without toxicity and with proven synergism between the properties of the material and silver. The results of the characterization analyzes demonstrate a potential material for application as a dressing.

[1] ABDEL-MOHSEN, A. M. et al., International Journal of Biological Macromolecules, v. 139, p. 730-739, 2019.

[2] ALAQD, K; SALEH, T. Journal of environmental analytical toxicology, v. 6. n.4, 2016.

Exploring Nucleotide-based Metal-Organic Frameworks for Quantum Dot Intracellular Delivery

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Nucleotides are effective ligands that can coordinate with metal ions, particularly transition metals, leading to the formation of metal-organic frameworks (MOFs). These nanoscopic aggregates have diverse biomedical applications, including intracellular drug transport, therapeutic molecule distribution, and bio-imaging. Quantum dots (QDs) are semiconductor nanoparticles with unique optical properties, making them suitable for combination with biocompatible nanostructures in biomedical applications. This study aimed to synthesize supramolecular complexes comprising guanosine-based nucleotides, metal ions, and graphene quantum dots, followed by detailed structural characterization and in vitro testing against tumour cell lines. A protocol was established for solubilizing guanosine monophosphate (GMP) in the presence of metal ions, such as Zn, Mg, Mn, Fe, Co, Cu, and Ni, along with graphene quantum dots. The nanoparticles results were analyzed using atomic force microscopy, scanning electron microscopy with energy dispersive spectroscopy (EDS) and steady-state fluorimetry. The complexes were also tested for cytotoxicity against HeLa cells, with successful production of ordered and crystalline GMP/QD nanoparticles observed in the presence of the aforementioned metals. Notably, MOFs prepared with Fe3+ were found to modulate the fluorescence capabilities of graphene QDs, leading to shifts in absorption/emission maxima or changes in fluorescence yield. The MOFs exhibited excellent tolerance by cells up to concentrations of mg/ml. Overall, this study presents a facile protocol for the development of nucleotide-based nanoparticles incorporating QDs, with promising applications in the development of biomaterials.

Acknowledgements: To FAPESP (grant n°19/20907-7) for financial support. To CNPq for a MSc. fellowship.

Exploring the Hydrogelation Behavior of a Designed Decapeptide for Cell Morphogenesis

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For this work designed nanostructured hydrogels using the sequence DGRL4DGW, a selfassembling peptide containing motifs from osteopontin (DGR) and fibronectin (DGW), two significant proteins involved in osteogenesis and cell adhesion. The resulting hydrogel was characterized using cutting edge biophysical techniques such as small angle neutron scattering under controlled temperature and atomic force microscopy. Furthermore, we explore the use of these matrices in promoting the growth of living cells and modulating the formation of three-dimensional cell spheroids by confocal microscopy and monitoring the viability of those cells along the time. The DGRL4DGW hydrogel was possible to be made by quick-gelation methods with small amounts of peptide (99% water and 1% of peptide), was thermostable and also maintained NIH3T3 and HeLa cells for 3 days in cell culture[1].

Acknowledgments
This work was supported by FAPESP under grants #2022/03056-6, #2019/19719-1 and #2021/10092-6 for financial support.

References

[1] L.R. de Mello, V. Carrascosa , E. Rebelato, M.A Juliano, I.W. Hamley, V. Castelletto, S.V. Vassiliades, W.A. Alves, C.R. Nakaie, E.R. da Silva. Nanostructure formation and cell spheroid morphogenesis of a peptide supramolecular hydrogel. Langmuir. 2022, 11;38(11) pp. 3434-45.

Gallium and niobium containing drug-loadable mesoporous bioactive glass nanoparticles

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Traditional osteosarcoma therapies tend to focus solely on eradicating residual cancer cells and often fail to promote local bone regeneration and even inhibit it due to lack of precise control over target cells, i.e., the treatment affects both normal and cancer cells [1,2]. An alternative for these problems is the use of bioceramics such as bioactive glasses (BGs) for targeted ionic therapy, through the release of ions that enhance osteogenic response and control micrometastasis [1]. In this work, we report the synthesis and characterization of sol-gel bioactive glasses containing niobium (Nb) and gallium (Ga), a multifunctional glass that has potentiating effects for bone repair and regeneration, concomitantly with a bone cancer targeted therapy. The synthesis is carried out via the sol-gel route coupled to self-propagating combustion method, developed by our research group [1]. The Ga3+ into the vitreous network promotes an increase in the network connectivity, contributing to an increase in the degree of polymerization of the glass, since part of the calcium ions that behave as network modifying agents were replaced by gallium ions that act as network formers. SEM microscopy for glass samples revealed a morphology and porosity structure characteristic of mesoporous bioactive glass (MBG) and also indicated the samples were meso-porous and formed by the stacking of nanometric plates, generating material with a high surface area and high porosity. These features are critical factors for the control of glass reactivity and bioactivity, which are very important for biomedical applications. Acknowledgments:The authors are grateful for the financial support provided by the São Paulo Research Foundation (FAPESP, 2022/03247-6). This research was carriedout at the Laboratory of Biomaterials and Biointerfaces (BBLab) of the ITA.References:[1] Medeiros, G. S et al. JNCS, 599, 121962 (2023). [2] Souza, L. ACS, 14, (40), 45156-45166 (2022).

Green-Emitting Phosphorus-Nitrogen Doped Carbon Dots: Enhanced Purification and Potential Application

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Carbon dots are a novel promising class of nanomaterials that have aroused growing attention due to their unique photoluminescence (PL) properties. These recently synthesized zero-dimensional nanomaterials can replace traditional semiconductor quantum dots, which exhibit significant toxicity and higher costs. However, most of the described carbon dots possess blue light emission under UV light, which is not suitable for cellular imaging and biological medium. Therefore, CDs with long-wavelength emission by nearly visible light sensitization attract enhanced attention. But successful fabrication of functional carbon dots is only half the battle. The application of CDs in modern technologies requires very small quantities of ultrapure CDs with precisely controllable size, shape, composition, and surface features. Recently, scientists have given more attention to the fact of the formation of molecular fluorophore species as by-products during CD synthesis. The presence of molecular fluorophores in the final sample leads to mistaken conclusions regarding the fundamental properties of these emerging nanoparticle thus, fluorophores are known to give rise to excitation-independent PL behavior. Therefore, a successful, efficient, affordable, and fast purification process before is needed to obtain reliable results on the properties of CDs. Essner et al. studied over 550 research articles and revealed that more than half of them used insufficient purification processes. Thus, the evaluation of the purification process of CDs is needed. In this study, we synthesized green-emitting phosphorus-nitrogen doped carbon dots via hydrothermal method from citric acid, ethylenediamine, and phosphonic or phosphoric acid as precursors. The reaction products were purified by two of the most traditional and commonly used methods - dialysis and preparative column chromatography. The performance of each method was evaluated and compared.

Green study of ZnO nanoparticles as photosensitizers in Photodynamic Therapy

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Photodynamic therapy (PDT) is a relatively new modality of disease treatment that has aroused interest in tumor therapy research. PDT involves the combination of three basic factors light sources, oxygen, and a photosensitizer (PS) [1]. PS requires being non-toxic to normal tissue in the dark and causing photodamage with an appropriate light source [1]. Studies show that the use of nanoparticles can increase the efficacy of PS in PDT, and zinc oxide nanoparticles (ZnO-NPs) are considered promising due to their unique properties. This study aimed at the green synthesis of ZnO-NPs for application as PS in PDT. The green synthesis process was carried out by microwave-assisted hydrothermal route using soybean extract as a reducing agent [2]. The ZnO-NPs were characterized by scanning electron microscopy, Fourier-transform infrared spectroscopy and X-ray diffraction, and used as a catalyst in the photodegradation assays of methylene blue dye, under black light. Photodegradation assays were carried out to test the effectiveness of the synthesized nanoparticles as photosensitizers. The XRD spectrum showed crystalline structures corresponding to ZnO, the FTIR showed the chemical absorption bands, and the morphology of the nanoparticles was confirmed by SEM. Thus, the preliminary results indicated the formation of ZnO-NPs and promising conditions for the application of these nanoparticles as photosensitizers for use in PDT.

Acknowledgements:The authors would like to thank the Postgraduate Program in Materials Science and Technology and Unesp Bauru for supporting research.

References:
[1] W. Yu et al. “A review and outlook in the treatment of osteosarcoma and other deep tumors with photodynamic therapy: from basic to deep”, Oncotarget, vol. 8, no 24 (2017).
[2] M. N. Alharthi et al. “Biosynthesis Microwave-Assisted of Zinc Oxide Nanoparticles with Ziziphus jujuba Leaves Extract: Characterization and Photocatalytic Application”, Nanomater. Basel Switz., vol. 11, no 7 (2021).

Green synthesis and characterization of zinc oxide nanoparticles using Tea tree essential oil

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Green synthesis of zinc oxide nanoparticles (ZnO NPs) involves the use of eco-friendly, non-toxic, and renewable sources, which has gained significant attention in recent years due to its potential applications in various fields. Some of the commonly used green synthesis methods for ZnO NPs include plants, microorganisms, and natural polymers. Tea tree essential oil is an aromatic oil that is derived from the leaves of the tea tree. Tea tree essential oil has been reported to have potential as a chelating agent and/or an oxidizing/reducing agent in the biosynthesis of NPs. In this study, ZnO NPs were synthesized using 0,15 M aqueous solution of zinc nitrate hexahydrate as a precursor, and 40g/L aqueous emulsion of tea tree essential oil containing Tween 80. The tea tree essential oil was added dropwise to the precursor solution, followed by the addition of a sodium hydroxide solution to adjust the pH to 8. The mixture was then stirred for four hours at 60°C, leading to the formation of ZnO NPs. The tea tree essential oil was found to be an effective reducing agent for the synthesis of zinc oxide nanoparticles, and the characterization carried out using Scanning Electron Microscopy (SEM) reveals the presence of zinc oxide nanoparticles in its agglomerated form resulting nanoparticles had a size range of 60-100nm. The average crystallite sizes of ZnO NPs calculated via X-rays diffraction analysis are found to be 25 nm. A UV-Vis absorption peak was observed at 380 nm, which is specific for ZnO NPs. FT-IR spectroscopic analysis shows absorption peak of Zn-O bonding between 442 and 512 cm-1. Overall, the use of tea tree essential oil in the synthesis of ZnO NPs is a promising approach that offers several advantages, such as eco-friendliness, low cost, and scalability. However, further research is needed to optimize the synthesis process and to evaluate the potential applications of these nanoparticles in various fields.

Green synthesis of gold nanoparticles with red propolis extract and its cytotoxic potential in mammalian fibroblasts

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The application of metallic nanoparticles has been explored in recent years due to their biocompatibility, adjustable optical properties and easily changeable surface chemistry. Studies with gold nanoparticles (AuNPs) have reported their functionality as drug carriers and molecules to improve diagnosis and treatment of diseases (BOTTEON, 2021). This work reports the green synthesis of gold nanoparticles with red propolis extract and its cytotoxic potential in mammalian fibroblasts (PVAuNPs). The development of ecologically correct processes has been increasing to optimize the production of PVAuNPs, using green technologies and contributing to the reduction of the environmental impact. This approach has been studied to replace methods commonly used in industry and thus reduce the cytotoxicity of these materials against cell lines (BANDEIRA, 2020). Propolis is a resinous product produced by bees, the variety of red propolis is obtained exclusively in northeastern Brazil and presented several biological activities such as antibacterial, anti-inflammatory, antioxidant, antitumor, among others (FROZZA, 2013). The cytotoxicity of gold nanoparticles biosynthed with red propolis ethanol extract was analyzed using the MTT assay in mammalian fibroblasts (L929). The preliminary results obtained showed that the nanoparticles had a spherical morphology with a varying between 40 and 60nm. Cell viability tests indicated absence of cytotoxicity in the L929 strain at concentrations up to 50 μg/ml, with an IC50 of 207.23 μg/ml. More studies are needed to determine the chemical and physical characterization of these biosynthed AuNPs, as well as to evaluate their antimicrobial and antifungal potential to better understand their applicability.

Gum arabic-gold nanorods composite effects on macrophages: towards tumor microenvironmentevaluation

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Macrophages are immune cells that play an important role in tumor microenvironment (TME). Gum Arabic (GA) is a well-known non-cytotoxic biopolymer that present immune modulation properties, and that has been used for nanoparticles synthesis and stabilization. In a previous work we have demonstrated in vitro gum arabic-gold nanords (GA-AuNRs) composite biocompatibility to fibroblasts, and their ability to reduce melanoma malignant characteristics. In vivo, GA-AuNRs impaired melanoma primary tumor growth in mice [1]. Now we investigate GA-AuNRs ability to activate macrophages towards TME modulation and melanoma progression impairment. The macrophage cell line RAW 264.7 was used to determine GA-AuNRs (from 1.8 ng/L to 18 mg/L) effects after 96 h, on cell viability (neutral red uptake), density (crystal violet), and nitric oxide (NO) production (Griess). GA was used as control and 3 independent experiments were performed. No statistically significant changes in cells viability and density were observed up to 0.18 mg/mL. NO production was not altered in the presence of non-cytotoxic GA-AuNRs concentrations. Primary macrophages from C57/BL6 mice bearing or not melanoma were used to determine GA-AuNRs anti-tumorigenic phenotype (M1) induction. All procedures using animals were approved by the Ethical Committee (SCB-UFPR CEUA № 1485). M1 phenotype was investigated by cells immunophenotyping, cytokines production (CBA kit), reactive oxygen species (ROS - DCF-DA assay), and phagocytosis activity in a preliminary study. The results suggest that M1 macrophage phenotype might be induced by GA-AuNRs, but further experiments need to be performed to validate it. In conclusion, in vivo TME modulation by GA-AuNRs composite could be occurring together with melanoma cells malignancy decrease. Acknowledgements:CAPES(PROAP-PPGBCM), PRPPG-UFPR(Edital apoio a pesquisa), CAPES and CNPq students fellowshipReferences:[1] J.P.Gonçalves et al., Int. J. Biol. Macromol. 185, 551-561 (2021)

Highly efficient ROS generation in carbon dots envisaging antimicrobial photodynamic therapy

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Over the past decade, several studies have investigated the viability of using carbon dots as photosensitizers in antimicrobial photodynamic therapy. The primary motivation is the substantial increase in the number of pathogenic strains resistant to antibiotics and disinfectants, which has caused an enhancement in mortality rates and health care costs. Therefore, many efforts have been made to develop new strategies to inactive deadly pathogens and to improve sterilization procedures. A remarkable attention has been devoted to photodynamic therapy with carbon dots as the photosensitizers. Like molecular photosensitizers, carbon dots may generate reactive oxygen species (ROS) upon photoexcitation in a specific wavelength. The main goal is the study of nanoparticles exhibiting an efficient intersystem crossing between singlet and triplet excited states, which may favor ROS generation through energy transfer to oxygen molecules dispersed in the carrier medium. Although several methods and precursors have been used, most carbon dots exhibit a low efficiency in ROS generation upon photoexcitation. In this context, the present study is devoted to investigate the physicochemical properties of carbon quantum dots (CD-MR) obtained from hydrothermal synthesis using methyl red as the precursor. CD-MR's time-resolved and steady-state fluorescence is investigated, considering the thermal effects on the photoluminescence. The possibility of using CD-MR to photogenerate reactive oxygen species (ROS) is also analyzed, where a high singlet oxygen quantum efficiency is verified. Moreover, it is studied the antimicrobial activity of carbon dots against bacteria (S. aureus and E. coli) and fungi (C. albicans and C. neoformans) strains upon photoexcitation at 532 nm. Using the susceptibility test of Kirby-Bauer, also labeled as the disk diffusion test, it is observed that such carbon nanoparticles present antimicrobial activity against gram-positive bacteria and fungi strains.

Hydrocolloid biobandage is produced from green synthesis using natural biopolymer and green propolisfor treatment of dermal lesions

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The concern for sustainability has driven medical research to search for alternatives to synthetic materials[1]. Developing effective biodegradable dressings that can treat skin lesions non-invasively and generate less waste is highly desirable. This work describes the green production of a biofilm-based on chitosan and albumin and its application as a carrier vehicle for the controlled release of propolis extract, for use as a hydrocolloid biobandage in the treatment of dermal lesions. In this study, a multifactorial optimization approach, involving core composite design and full-factor design (2 3), was used to evaluate the main factors that influence biofilm formation and optimal synthesis conditions. The factors evaluated were (i) mass of chitosan and albumin (0.5-2g), (ii) concentration of acetic acid (1-5%, w/v), and (iii) the presence or absence of a cross-linking agent (glutaraldehyde, 0-0.5%). As a result of this optimization, it was observed that using (i) a mass of chitosan (1.5g); (ii) the concentration of acetic acid (2.0%, m/v); and (iii) the absence of glutaraldehyde allowed to produce a resistant biofilm with a high degree of tumescence, enabling a high load capacity of the drug. The biofilm generated under optimal conditions was characterized using specific characterization techniques (SEM, FT-IR, DRX, and TGA). In vitro studies to assess (i) drug loading capacity, (ii) controlled drug release, (iii) membrane permeation, and (iv) toxicity are ongoing and will be presented at the event.

Acknowledgements: FAPEMIG (APQ-01786-22; RED-00144-22); CNPq (405828/2022-5) and CAPES

References: [1] J.Baranwal et al., Polymers vol. 14, 983, (2022).

Impedimetric biosensor for tuberculosis diagnosis

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Tuberculosis (TB) is a mycobacterial infection commonly caused by the etiologic agent Mycobacterium tuberculosis. Reported over a century ago, TB was the leading cause of death from a single infectious agent until the coronavirus (COVID-19) pandemic, ranking above HIV/AIDS [1]. The traditional methods for TB diagnosis, i.e. sputum smear microscopy, liquid culture, and recently, molecular tests, present many limitations, such as the low sensitivity and long periods of time to obtain the results, frequently leading to incorrect diagnosis and unnecessary treatment [2]. Electrochemical sensors have attracted attention due to their simplicity, low-cost instrumentation, very low detection limits, and rapid response [3]. In this study we present an electrochemical impedimetric immunosensor for the detection of recombinant protein CFP10:ESAT6 forthe diagnosis of M. tuberculosis. The immunosensor was developed onto indium tin oxide (ITO) electrodes modified by 3-aminopropyltrimethoxysilane (APTES) monolayer to covalently immobilize anti-CFP10 antibodies. The protein interaction with the antibody recognition platform was directly monitored and measured by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The immunosensors were characterized with atomic force microscopy (AFM) and Raman spectroscopy. After the analytical features optimization, a linear response from 0.5 ng ml-1 to 50 ng ml-1 of pCFP10:ESAT6, a limit of detection (LOD) of 4.80 ng ml-1 and a limit of quantification (LOQ) of 15.97 ng ml-1 were achieved, in a 4-hour essay time. The results indicate that the immunosensor is suitable, accurate, and selective for CFP10:ESAT6 protein detection, presenting exquisite features for point-of-care diagnostic applications in the future.

In Situ SAXS/UV-vis Nucleation and Growth of Gold Nanoparticles in HEPES buffer solution

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The research about the mechanisms of gold nanoparticles synthesis has received great attention in scientific research because their wide variety of applications for biomedical treatments, such as: clinical diagnostics, disease prevention and drug delivery. One of the biggest challenges in the AuNP synthesis is the control of colloidal stability and monodisperse size distribution. In fact, the AuNPs regarding size, shape, size dispersity is determined since the first steps of gold ion reduction and nanocluster nucleation. In these steps the role played by gold ion hydration is crucial and not completely understood. The present study has a main goal to understand the role played by gold ion and gold nanocluster hydration in the process of gold nanoparticles synthesis. The strategy was to investigate the synthesis of gold nanoparticles in H2O and D2O, using 0.45 mM HAuCl4 and HEPES (0.25 and 30 mM) as reducing agent, in phosphate buffer pH 10. The synthesis was carried out by chemical route, without the addition of polymeric surfactants and additives that interfere in the morphology of the nanostructures. The isotopic substitution of the solvent clearly affected the properties of AuNPs. The specific structural, morphological, and optical properties of AuNPs synthesized in H2O and D2O were characterized in situ by ultraviolet-visible spectroscopy (UV-vis) and small angle X-ray scattering (SAXS) as a function of time and temperature. The results allow us to develop a model to describe the mechanisms underlaying the gold nanocrystal growing influenced by the solvent for a better control of AuNP synthesis.

Investigating Drug-Polymer Interactions of QPhNO2, PCL, PLGA, and PMMA using DFT Quantum Chemical Calculations for nanoencapsulation Applications.

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This study investigates the interaction between QPhNO2 drug and polymers (PMMA, PCL, PLGA) for nanoencapsulation. The aim is to predict drug release factors and determine the optimal drug-polymer pair. Using DFT calculations, it is found that QPhNO2 interacts more favorably with PLGA than PCL. Molecular docking simulations and ab initio calculations predict molecular properties and therapeutic potential[1], highlighting computational chemistry''s value in drug development. Methods include Spartan''14 software and DFT approach (B3LYP, 6-311++G** basis set)[2] for molecular interaction analysis. ArgusLab 4.0.1 and Software Discovery Studio Visualizer support simulations. This research contributes to drug design and enhancement. QPhNO2, a potential anticancer drug, has limited clinical use due to solubility issues. Nanoencapsulation is promising for cancer drug administration using PCL, PLGA, and PMMA materials. Through computational methods, the study characterizes QPhNO2''s interaction with each polymer, predicting release factors and identifying the best pair. The results inform nanoparticle structure, impacting release mechanisms, and empha computational chemistry''s role in drug development and molecule characterization.

Investigating the Biodistribution of Magnetic Nanoparticles in Rat Models of Cirrhosis-Induced Hepatocarcinogenesis

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Since magnetic nanoparticles (MNPs) presented the possibility of being used to liver diseases diagnose and treatment in recent years. This study aimed to inquire the influence of cirrhosis in the liver function. The distribution profile of MNPs was monitored by a real-time image acquisition approach after intravenous administration. Biodistribution profile was assessed based o Alternate Current Biosusceptometry (ACB) images obtained over regions of interest (ROIs) selected in heart and liver position according to previously decided anatomical references. The obtained signals allowed the quantification of pharmacokinetic parameters, inquiring that hepatic uptake was compromised during cirrhosis, which influences blood exchange between liver parenchyma and circulation. Kupffer cells remained constant during cirrhotic period, so the capillarization of hepatic sinusoids was considered responsible for the change in liver function.

Investigation of the electrospinning technique in obtaining nanofibers: synthesis, standardization and characterization

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Electrospinning is a methodology that uses a high voltage in obtaining nanofibers. In our study, the following parameters were observed: voltage of 15kV and 23kV, needle diameter of 0.7mm and 1.2 mm and distance to the 12 cm collector, in obtaining nanofibers of silver nanoparticles with red propolis from Alagoas (NPAg-RPA). Being the polymer matrix selected for the work, polycaprolactone (PCL), in silver concentrations of 1%, 2% and 5%. The characterization was performed by SEM, PXRD, TG/DSC, FTIR. Evaluating the applied voltage, the average diameters for the voltages 15 and 23 kV were found, being chosen the voltage of 15kV, for presenting better aspect in the diameter of the fibers obtained. The diameter of the needle at 1.2 mm was more efficient, providing a shorter time of contact of the solution with the atmosphere and presence of moisture. By SEM it was evaluated that higher concentrations of NPAg configured a reduction in fiber diameter and a greater uniformity of distribution. By PXRD, peaks related to PCL were observed both in its pure form and in the products associated with NPAg-RPA, as well as crystalline planes related to NPAg. By TG it was demonstrated that there were no changes in the decomposition of the products compared to the pure polymer. At the end, it was observed that the best parameters were voltage of 15kv and needle diameter 1.2mm. Through characterization techniques, it was observed that the nanofibers obtained demonstrate aspect ideas for a dressing, being a promising agent for research.

[1] BAYAT, S. et al. Bromelain-loaded chitosan nanofibers prepared by electrospinning method for burn wound healing in animal models. Life Sciences, v. 229, n. April, p. 57-66, 2019.

[2] BOZKAYA, O. et al. Production and characterization of hybrid nanofiber wound dressing containing Centella asiatica coated silver nanoparticles by mutual electrospinning method. European Polymer Journal, v. 166, p. 111023, 2022.

Investigation of the formation of protein corona in bismuth sulfide nanoparticles

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Cancer is one of the main threats to human health due to the large number of cases and high mortality rates. Although radiotherapy is the main course of treatment, it is not specific for tumor cells, affecting also healthy tissues. Radiosensitizers provide a novel and simple solution to this problem, since they increase the amount of radiation that a cell can absorb. Bismuth sulfide (Bi_2S_3) nanoparticles (Np) have interesting properties to be used as radiosensitizers and could be accumulated in tumors by EPR effect. However, the interaction between Np and blood compounds, resulting in the protein corona formation, can produce a Np stability loss, which influences the final treatment. In this study, we synthesized Bi_2S_3Np using a hot injection method and oleylamine as ligand, following a ligand exchange process. TEM images showed rods measuring on average 4.2nm in width and 27.4nm in length. The ligand was successfully exchanged by polyvinylpyrrolidone (PVP) or polyethyleneglycol (PEG), confirmed by Diffuse FTIR and TG-DSC analyses. The Bi_2S_3-PVP and Bi_2S_3-PEG hydrodynamic size were 142.1 nm and 124.4 nm, determined by DLS, and their Z potential were -5.24mV and -1.15mV respectively. To study the protein corona formation, Bi_2S_3-PVP or Bi_2S_3-PEG Np were incubated in cell culture medium DMEM supplemented by 10% FBS (DMEM-FBS) and artificial blood plasma fluid (aBPF), for 15 min to 48 h at 37 ℃. According to the hydrodynamic sizes and Z potentials, the Np-protein corona assembly was stable during the incubation time. The average size in DMEM-FBS increased to 206.7 nm and 180.6 nm and the Z potential decreased to -11.8mV and -20.4mV for Bi_2S_3-PVP and Bi_2S_3-PEG Np, respectively. In aBPF, the size was 160.1 nm and 149.8 nm and the Z potential was -11.8mV and -15.0mV for the same Np. Our findings demonstrate that the protein corona formation enhances the ability of the Np to interact with cells in vitro and to move through the bloodstream for further applications in vivo.

LIPID TAIL DEPENDENCE ON THE EFFECTS OF THE PESTICIDE ETHEPHON IN PHOSPHATIDYLCHOLINES MODEL MEMBRANES

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Ethephon is a pesticide largely used in Brazilas a growth regulator, and in fruit maturation. Studies related to the long-term exposure effects of the ethephon in humans through water and food ingestion are sparse and inconclusive. For this reason, we investigated the effects of the ethephon in Langmuir monolayer and lipid bilayers (LUVs and GUVs) as bioinspired c system of the plasma cell membrane, since this organelle is responsible for maintaining cell integrity and actsas a selective barrier to most external agents. The phospholipids used DPPC, POPC and DOPC differ in terms of the number of unsaturation present in the tail of hydrocarbons (0, 1 and 2, respectively) and were chosen because they belong to the phosphatidylcholine group, majority present in most cell membranes. Ethephon interacts at the air/water interface with the POPC and DOPC, causing displacement in the isotherms. The in situ infrared reflection absorption spectroscopy with polarization modulation (PM-IRRAS) spectra indicated changes in choline and phosphate head groups of the lipids in presence of ethephon. However, the band referring to stretching of the unhydrated carbonyl group (C=O) is displaced only for POPC. This phospholipid is sensitive to hydrolysis reactions precisely with bond breakage in the carbonyl group. Ethephon, in turn, seems to be favoring this breakdown, much more evident than the peroxidation reactions commonly seen for DOPC. Ethephon reduced the Zeta potential of the POPC LUVs, therefore confirming preferential interaction in the polar head group moiety of the lipids. Microscopy images revealed that only POPC GUVs suffered considerable damage in the presence of ethephon, causing loss of phase contrast as well as vesicle bursts. This work highlights the importance of understanding the behavior of ethephon in structures that preserve chemical structure of the cell membrane, being good indicators of its action in biological processes.

Acknowledgments: FAPESP, CNPq, INEO, CAPES

Achillea millefolium: Biogenic Silver Nanoparticles (AgNPs) to control pathogenic microorganisms.

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Biogenic nanoparticles have less residual toxicity, better stability, and the possibility of size control. They feature a coating of proteins and metabolites (capping) derived from the organism used in the process, and this capping contributes to the stability and biological activity. The plant *Achillea millefolium*has shown to be a promising reducing and stabilising agent in the biogenic synthesis of silver nanoparticles (AgNP) with antimicrobial potential and low toxicity, being a possible source of new drugs. The synthesis of biogenic AgNPs was characterised using dynamic light scattering - DLS and nanoparticle tracking - NTA technique their cytotoxic potential (MTT Test) and their activity in controlling pathogens (Disc diffusion and Minimum Inhibitory Concentration - MIC) were also evaluated. AgNPs had a polydispersity index of 0.565 ± 0.006, a zeta potential of -23.4 ± 0.5 mV, a mean sizeof 77.2 ± 0.9 nm and a concentration of 7.38 x 1010 NPs/mL. MTT did not show IC50 valuesin any strain, indicating low toxicity. The disk-diffusion test and MIC showed the antimicrobial potential of AgNPs on the tested microorganisms. Therefore, they may be a viable option for application in the control of pathogens, with the absence of cytotoxic effects at effective concentrations, aiming at their future application in the health area. New studies must be carried out to obtain a safe final material.

Acknowledgements: Universidade de Sorocaba e Unesp de Sorocaba.

References: [1] LOGESWARI, P.; SILAMBARASAN, S.; ABRAHAM, J. Synthesis of silver nanoparticles using plant extract and analysis of their antimicrobial property. Journal of Saudi Chemical Society, v. 19, p. 311-7, 2015.

In vivo Evaluation of a Catalyst-free Click Chemistry Hydrogel for Skin Tissue Regeneration

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The pursuit of ideal biomaterials that perfectly match the building blocks of organs and tissues and that are well recognized and integrated with the microenvironment surrounding the tissues and cells is a never-ending challenge[1]. CS provides the tissue with a good resistance to compression and exhibits anti-inflammatory and anti-catabolic activity. Present in large quantities in native ECM and on the surface of cells, this polymer enhances growth and contributes to neurotrophic factors. CS is an important component in wound healing, promoting cell adhesion and cell proliferation[2]. Herein, we disclose a versatile and facile catalyst-free click chemistry for the generation of an innovative hydrogel by combining chondroitin sulfate (CS) and polyethylene glycol (PEG). The hydrogel demonstrated not only enhanced blood perfusion and an increased number of blood vessels, but also desirable fibrous matrix orientation and normal collagen deposition. Taken together, these results demonstrate the potential of the hydrogel to improve wound repair and hold promise for in situ skin tissue engineering applications.

Acknowledgements:

The authors acknowledge support from CAPES (scholarship), Serrapilheira institute (Serra-1709-19479 to AOL) and CNPq (442731/2020-5 and 404683/2018-5), and Research Fellowship.

References:

[1] Uludağ, H. Grand Challenges in Biomaterials. Front. Bioeng. Biotechnol. 2014.

[2] Bhowmick, S.; Thanusha, A.V.; Kumar, A.; Scharnweber, D.; Rother, S.; Koul, V. Nanofibrous artificial skin substitute composed of mPEG-PCL grafted gelatin/hyaluronan/chondroitin sulfate/sericin for 2nd degree burn care: In vitro and in vivo study. RSC Adv. 2018.

Magnetic Nanoparticle Biodistribution and Clearance:A Long-Term Study Using AC Biosusceptometry

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After the administration of magnetic nanoparticles (MNPs) in an organism, several parameters including retention and elimination profiles, physiological parameters and possible interactions must be assessed. Alternate Current Biosusceptometry (ACB), a biomagnetic detection system, can be used to detect and quantify MNPs. The study aimed to evaluate MNPs clearance and biodistribution profiles over long-time in vivo analysis and determine the elimination time, accessed by the association between ACB and manganese ferrite (MnFe2O4) nanoparticles. Samples of liver, lungs, spleen, kidneys, heart, and blood were collected over 60 days for biodistribution and elimination analysis. During the assessment period animal's feces were also collected. It was possible to notice a higher uptake by liver and spleen due to intrinsic retention and uptake factors. Over these 60 days we observed an absence of MNPs in spleen and significant decay in the liver. It was also possible to determine the MNPs half-life trough by the liver and spleen elimination. The results showed a decay on the concentration profile over the time, which suggest that in addition to elimination by feces, there is an endogenous mechanism of agglomeration or metabolization involved, resulting in loss of the signal intensity.

Melatonin loaded polymeric nanoparticles for glioblastoma therapy

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Glioblastoma (GBM) is a highly malignant brain tumor with an elevated risk of recurrence and mortality. Current treatment options, s Current treatment options, such as a therapeutic regimen applying temozolomide (TMZ), show limited efficacy due to the invasive and heterogeneous nature of the tumor and the development of resistance mechanisms[1]. Melatonin (MLT) has shown promising anticancer activity in GBM, due to its ability to reduce the tumor cell proliferation, and modulate different signaling and biological processes.[2]In this study, we developed a new approach for GBM treatment by encapsulating MLT in poly(lactide-co-glycolide) (PLGA) nanocarriers. These nanosystems have shown an encapsulation efficiency of 94%, a diameter of 112 ± 5 nm, a polydispersity index (PDI) of 0.164 ± 0.015, and a zeta potential of -14 ± 2 mV, which are suitable physical properties fordrug delivery intended nose-to-brain approach. Our novel anticancer system may offer a promising therapeutic opportunity for GBM treatment.

Acknowledgments: This work was supported by the National Council for Scientific and Technological Development (CNPq), Grant 381475/2022-0

References:
1 doi:10.1016/j.gendis.2016.04.007
2 doi:10.3390/cells9030599

Modulation of the photoacoustic signal of AuNRs byLED or Laser: fundamental studies for theranostic applications.

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Gold nanoparticles in the form of nanorods (AuNRs) have shown promising potential as contrast agent in photoacoustic imaging due to their localized surface plasmon resonance (LSPR) which allows them to efficiently absorb light in the near-infrared region. However, it is crucial to evaluate the impact of commonly used emitters, such as LEDs and lasers, on photoacoustic signal intensity a structural property of AuNRs. To this end, AuNRs of different lengths were synthesized and characterized by spectroscopies, dynamic light scattering, nanoparticle tracking analysis, and transmission electron microscopy. The photoacoustic signal was evaluated for different particle sizes and concentrations using both LED and laser emitters. The experimental results revealed that the intensity of the photoacoustic signal was dependent on both length and concentration of AuNR. In addition, significant morphological changes were observed upon irradiation of AuNRs with a laser, as compared to LED. This study provides new insights into the signal parameters of AuNRs in photoacoustic imaging and their interaction with AuNRs during irradiation. Notably, the study suggests that LEDs may have advantages as emitters for photoacoustic imaging using AuNRs as contrast agent.

Acknowledgements: CNPq and Fapesp grants: 2021/14775-0.

References:

[1] Kumar, D., Soni, R. K., Ghai, D. P. (2020). Pulsed photoacoustic and photothermal response of gold nanoparticles. Nanotechnology, 31. https://doi.org/10.1088/1361-6528/ab47ae

[2] Hariri, A., Lemaster, J., Wang, J., Jeevarathinam, A. S., Chao, D. L., Jokerst, J. v. (2018). The characterization of an economic and portable LED-based photoacoustic imaging system to facilitate molecular imaging. Photoacoustics, 9, 10-20. https://doi.org/10.1016/j.pacs.2017.11.001

Monolayer magnetic nanospheres have selective binding that allow the concentration of low-abundance proteins from blood serum

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The efficiency of magnetic nanosphere (Fe304) coated with L-histidine (L-his) was evaluated to enrich proteins of low-abundance in human serum that are potentials biomarkers of human disease. The chemometric analysis provided optimal conditions to concentrate low-abundance proteins, reducing the dynamic range of protein levels in the serum sample. Three independent factors (temperature, mass ratio and pH) were tested. The optimal condition achieved was using temperature (42°C), ratio (3:1, nanomaterial/sample) and pH (5.5). The kinetic study revealed that the adsorption of proteins on the surface of the monolayer magnetic nanosphere follows a pseudo-second order model with R2=0.9153. Adsorption isotherm experiments revealed high capacity of protein adsorption on the surface of the nanosphere, indicating a maximum theoretical adsorption capacity (qmax) of 123.45 mg g-1. The efficiency of enrichment of low-abundance proteins in the serum sample was evaluated by gel electrophoresis (SDS-PAGE). Furthermore, when compared to the method using organic solvent (acetonitrile), Fe304@L-his showed adequate efficiency to simplify the serum sample. Using MALDI-TOF/TOF, approximately 50 proteins were identified using Fe304@L-his. As a proof of concept for this study, patients with bipolar disorder and healthy controls were discriminated using the magnetic nanosphere as an adsorbent in the sample preparation step. These results show that Fe304@L-his can be a fast, robust, and simple alternative to enrich low-abundance proteins in human serum, emerging as an important strategy in the search for biomarkers of human diseases.

Acknowledgements:FAPEMIG (APQ-01786-22; RED-00144-22); CNPq (405828/2022-5) and CAPES

References:[1] P.E. Geyer et al. Mol. Syst. Biol. vol. 13, 942, (2017).[2] J.Paul and T.D. Veenstra, Separations vol. 9 (2022).[3] J.R de Jesus et al. Talanta vol. 170, 199-209 (2017).

Multifunctional magnetic nanoparticles coated bynatural rubber latex for theranostics applications

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Magnetic nanoparticles (MNPs) have broad utilization in biomedicine, such as drug delivery, magnetic hyperthermia (MH), and as contrast agents for different imaging modalities. However, their effectiveness as a contrast agent for ultrasound and photoacoustic imaging (PAI) is limited. This study aimed to evaluate the effectiveness of coating magnetic nanoparticles with natural rubber latex (NRL) to overcome this drawback. Furthermore, the impact of NRL coating on MNPs'' heating mediator potential in MH was investigated. MNPs were synthesized via the coprecipitation method, utilizing three NRL coating volumes (0, 100, and 400 μL) [1]. The MNPs were submitted to several diagnostic techniques, such as magneto-motive ultrasound (MMUS) and photoacoustic imaging, in addition to being used in magnetic hyperthermia. The average size of the MNPs ranged from 10 to 20 nm, as confirmed by X-ray diffraction and transmission electron microscopy. Magnetic hysteresis curves indicated superparamagnetic samples. NRL-coated MNPs showed higher saturation magnetization, improving MMUS displacement, and increased optical absorbance, resulting in improved PA image quality. SNR values for the PA images were 261, 481, and 650 for uncoated MNPs, NRL-100, and NRL-400, respectively. For MH experiments, Specific Loss Power values of 57, 43 and 40 W/g were obtained using 0.25% (wt.) of MNPs coated with 0, 100 and 400 μL of NRL. These findings suggest that NRL-coated MNPs are promising candidates for contrast agents in both MMUS and PA imaging, as well as heat mediators for MH in tumor treatment.

Acknowledgments:
The authors would like to express their gratitude to the University of São Paulo, FAPESP 2021/06728-2, 2013-18854-6, CNPq and CAPES code 001.

References:
[1] Arsalani, Soudabeh et al. Green Synthesis and Surface Modification of Iron Oxide Nanoparticles with Enhanced Magnetization Using Natural Rubber Latex. ACS Sustainable Chemistry Engineering. 2018 6 (11), 13756-13765.

Multimodal nanosensors with optical and paramagnetic properties based on hydrophilic $AgInSe_2$ quantum dots

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Magnetic resonance imaging (MRI) is one of the current imaging techniques for cancer diagnoses. Despite having high resolution, it has low sensitivity. This limitation can occur even when contrast agents (CAs) are used. In these cases, the concentration of the CAs is not enough to show differentiation between healthy and pathological tissues [1]. One strategy that has been used in the past years is the association of CAs molecules to nanoparticles, such as quantum dots (QDs). QDs usually have a typical size ranging from 2 to 10 nm and unique optical properties. Besides, they have active surface for (bio)conjugation, allowing the attachment of CAs and biomolecules. Folic acid has been used for specific targeting of cancer cells due to the overexpression of folate receptors on most of these cells [2] In this work, we prepared AgInSe2 QDs in an aqueous medium, using a one-pot method and mercaptosuccinic acid as the stabilizer. These QDs were conjugated to thiolate DOTA complex derivatives and folic acid molecules, through dative bonds. The preliminary results showed a loss of around 50% of the emission intensity of the QDs after the conjugation procedures. And relaxometric studies showed an increase in the relaxivity values of these nanosensors, compared with the free Gd-DOTA complex in solution. Nevertheless, the systems developed show potential to be used as CAs for cancer diagnosis.

Acknowledgments:

The authors thank CAPES, CNPq, FACEPE (IBPG-0985-3.03/20), UFPE, and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020) for financial support.

References:

[1] G. M. Albuquerque, I. Souza-Sobrinha, S. D. Coiado, B. S. Santos, A. Fontes, G. A. L. Pereira, G. Pereira. Topics in Current Chemistry, 379, 12, 1-35, 2021.

[2] C. A. P. Monteiro, A. D. P. R. Oliveira, R. C. Silva, R. R. M. Lima, F. O. Souto, M. O. Baratti, H. F. Carvalho, B. S. Santos, P. E. Cabral Filho, A. Fontes. Journal of photochemistry and photobiology. B, Biology, 209, 111918, 2020.

Nanoparticles based on amphiphilic pullulan-g-poly(Z-L-lysine) and amphiphilic cationic pullulan-DEAE-g- poly(Z-L-lysine)

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Amphiphilic polymers have been attracting great interesting in pharmaceutical formulations due to their self-aggregation ability, which can promote nano or microparticles systems in an aqueous medium [1]. Poly(Z-L-Lysine) (PZLL) is a polypeptide in which the presence of aromatic carbamate (Z) can result in a conjugation with lipophilic drugs, and contribute for improving the polysaccharide structing making it appropriate for biomedical applications [2]. The presence of a cationic group in a copolymer can promote the sensibility to a pH change under solution, resulting in controlled release in drug delivery systems [2]. In order to compare the amphiphilic copolymers with and without the cationic properties, the amphiphilic pullulan was prepared by two different routes, first it was reacted with 2-Chloro-N,N-diethylethylamine (DEAE), resulting in PULL-DEAE, followed by the ring opening copolymerization (ROP) of carbobenzyloxy-l-lysine N-carboxyanhydride (Lys(Z)- NCA), obtaining PULL-DEAE-g-PZLL. The same copolymerization was applied, however to unmodified pullulan, resulting in the second amphiphilic copolymer, without the cationic group, PULL-g-PZLL. The final product was nanoprecipitated by dialysis method and the particles were evaluated by, particle size, particle distribution, zeta potential and sensibility under different pHs. The polymers resulted can be applied in the biomedical field, in different areas, according to the final properties obtained, besides, it can be used to encapsulate different actives.

Acknowledgements: FAPESP (2019/12940-4)

References: [1] M.Nichifor, M.C.Stanciu, B.C.Simionescu, v. 82, p. 965-975. 2010.
[2] L.T.Carvalho, A.J.R.M.Teixeira, R.M.Moraes, R.F.S.Barbosa, R.C.Queiroz d , D.B.Tada, D.R.Mulinari, D.Rosa, M.I.Ré, S.F.Medeiros, v. 181, 105441, 2022.

Nanoparticles containing carotenoids reduce foodintake and improve hepatic retinol

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Obesity is a chronic disease characterized by excess body fat associated with subclinical inflammatory responsible for damages, such as micronutrient deficiencies such as retinol [1]. Beta-carotene is a precursor of retinol but is unstable to environmental conditions and can be preserved and/or enhanced functionality by nanoencapsulation [2]. The study evaluated the effect of a crude extract rich in β-carotene from Cantaloupe melon (Cucumis melo L.) (CE) nanoencapsulated (EPG) on food intake, weight, and hepatic retinol in Wistar rats with diet-induced obesity. CE was nanoencapsulated in porcine gelatin by O/W emulsification technique and characterized by SEM, Laser Diffraction, FTIR, and carotenoid encapsulation efficiency (EE) determine whether the new batch obtained was as in previous studies. Wistar rats with obesity induced by a high glycemic index and high glycemic load diet (HGLI diet) were divided into three groups (n = 5): HGLI diet + water, HGLI diet + CE (12.5 mg/kg), and HGLI diet + EPG (50 mg/kg containing 0.62 mg/ml of CE) administered by gavage (1mL) for ten days evaluated for dietary intake, weight, and hepatic retinol. EPG presented a smooth surface, mean diameter of 80 (8.59) nm, and polydispersion index of 0.47 (0.09), new chemical interactions, and EE of 95.20% (0.74), as those obtained in previous studies. Animals treated with EPG showed a lower dietary intake ($p < 0.05$) and no significant change in the animal's weight in the groups. EPG-treated group had higher hepatic retinol ($p < 0.05$) [266 (45) μg/g] than the untreated [186 (23.8) μg/g] and treated with CE [175 (8.08) μg/g]. Nanoencapsulation promoted protection and contributed to the bioactive compound's absorption, allowing a higher concentration of retinol in the liver of obese animals.

Acknowledgment: National Council for Scientific and Technological Development.

References:

[1] M.A Beydoun, et al. Nutr Rev., 77, 32-45 (2019).

[2] J.L.C. Queiroz, et al. Food Chem., 385, 132593 (2022).

Nanostructured Materials Based on the Self-Assemblyof the Antidiuretic Peptide Desmopressin

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Desmopressin acetate (DDAVP) is a synthetic analog of the antidiuretic hormone vasopressin largely employed as a first-line pharmacological approach for urinary diseases such as diabetes insipidus and nocturia. Herein, we aimed to develop nanostructured materials with antidiuretic properties based on the self-assembling capabilities of DDAVP. In addition, we provide information about its structure, from the molecular level to the nanometer range. Also, we investigate the nanoparticle' s cytotoxicity effects. The structural analyses were performed through spectroscopic techniques (fluorimetry and circular dichroism), whereas morphology assays were carried out using atomic force microscopy. The cytotoxicity was determined by the MIC value. DDAVP was found to form nanometric aggregates at critical concentrations near to 1 mg/ml, with β-sheet conformations dominating the secondary structure of the aggregates. Quenching of tyrosine emission indicated a major role of hydrophobic domains in the self-assembly. The interaction of peptides with lipid membranes was also demonstrated, showing that their secondary structure is modified in the presence of membranes, and interaction with lipid bilayers being driven by hydrophobic domains. Cytotoxicity was not observed even in high concentrations above the CAC, which did not influence in the microorganisms growth. We demonstrated the fabrication of ordered nanoassemblies based on desmopressin, which is stabilized by β-sheets. It was also verified that the nanoparticles interact with lipid membranes, suggesting they can associate to cell membranes. The nanoparticles were not cytotoxic, showing its potential biomedical use. To our knowledge this is the first study reporting the production of nanostructured materials from peptides endowed with antidiuretic activity.

Acknowledgements: To FAPESP (grant nº#2022/03056-6) for financial support. To CAPES (finance code 001) for fellowships.

Nanotheranostic systems as a new strategy for the combined treatment of drug-resistant tuberculosis

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Several strategies have been used to overcome the therapeutic limitations of Resistant tuberculosis (TB) and multidrug-resistant TB, however, issues related to lack of effectiveness are still a concern [1]. As an alternative, nanotheranostic systems have been used to modulate the resistance of some TB drugs, which is possible due to their reduced that allows for different delivery mechanisms when compared to free drug. In addition, the use of theranostic nanosystems allows the combination of different therapies, such as the use of a radiation source to promote photothermia. In this study we developed a nanotheranostic system for TB therapy based on the encapsulation of standard anti-tuberculosis drugs, isoniazid and rifampicin, in a PLGA system containing AuNRs to promote phototermia (named PLGA/ISO-RIF/AuNRs). The physicochemical analysis revealed a spherical morphology with hydrodynamic of 253 ± 9.3, a polydisperse index (PdI) of 0.167 ± 0.044 and a potential zeta of +32 ± 3 mV. The encapsulation efficiency was estimated at ca. 75% for both drugs. PLGA/ISO-RIF/AuNRs nanoparticles were administrated in murine (BMDM) and human (THP1) macrophages showing synergy in the results compared to PLGA NP or PLGA/ISO-RIF NPs.

Acknowledgments: Financial support from CNPq is gratefully acknowledged.

References:
[1] J. Ghitman, E. Iuliana Biru, Review of hybrid PLGA nanoparticles: future of smart drug delivery and theranostics medicine, Materials Design vol. 193, (2020)

Nile Tilapia Skin Impregnated with Antibacterial Silver/Titanium Dioxide Compounds

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The development of alternative (and antibiotic-free) antibacterial and antibiofilm agents is an important strategy to prevent bacterial resistance to antibiotics. With this aim, the production of mixed oxides has been explored by incorporating silver nanoparticles in titanium dioxide as an antibacterial agent with behavior dependent on the concentration of silver, for the following step of incorporation into the skin of Nile tilapia [1]. Three experimental systems were evaluated: pure titanium dioxide (TiO_2), TiO_2 with 2% by weight of $AgNO_3$ precursor (TiO_2/Ag2%) and TiO_2 with 10% by weight of $AgNO_3$ precursor (TiO_2/Ag10%). The antibacterial and antibiofilm activity was tested against the Staphylococcus aureus strain (ATCC 25923) which is a biofilm-producing bacteria. The influence of silver concentration was confirmed through the results of microdilution tests in broth, kill time curve and diffusion in agar, in which the pure titanium dioxide (TiO_2) did not show antibacterial activity, but the TiO_2/Ag 2% sample exhibited a minimum bactericidal concentration (MBC) of 6.25 mg/mL, kill time in the order of 45 min and inhibition halo of 21.6 mm; and the sample with the highest silver content (TiO_2/Ag10%) had a MBC of 3.13 mg/mL, characteristic kill time of 30 min and inhibition halo of 23.81 mm. In addition to showing the best results in terms of antibacterial activity, samples prepared with high concentrations of silver (10% by weight of the $AgNO_3$ precursor) showed a good antibiofilm response. These results indicate that tilapia skin modified with mixed oxides acquires antibacterial behavior and can be exploited as a xenograft.

Paclitaxel encapsulated in PLGA nanoparticles for lung cancer therapy

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Lung cancer is a type of cancer that has a high occurrence and fatality rate in Brazil and globally. It is usually detected in advanced stages, which makes its prognosis difficult. In this context, researchers have explored novel proposals using nanotechnology to enhance the effectiveness and bioavailability of drugs for treating this type of cancer. In this studywe have developed a nanostructured system from poly(lactic acid-co-glycolic acid) (PLGA) to release paclitaxel (PTX) in the treatment of lung cancer to modulate or enhance the action of this drug. We used a simple emulsion technique to develop polymeric nanoparticles (NPs) containing PTX. The NPs had an average diameter of 162 nm, polydispersity index (PDI) of 0.168 in, and zeta potential of -23 mV. Further physicochemical analyses are underway to improve the characterization of the NPs before subsequent viability and cellular internalization assays. The development of these nanosystems is significant in the pursuit of suitable therapeutic approaches for managing lung cancer.

Photodithazine-nanoclay composites to improve antimicrobial activity.

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Advanced nanomaterials for drug delivery systems are the main focus of numerous research groups and, its application on drug resistance problem is probably the biggest challenged faced by scientists in the field. Indeed, in 2018, WHO declared the antimicrobial resistance as ‘(…) one of the top 10 global public health threats facing humanity’, along with a similar problem in cancer treatment: the chemotherapeutic drug resistant cells. In both cases, photoinactivation systems [1] and photodynamic therapy (PDT) can overcome the problem of drug resistance when compared to the traditional approaches. For PDT, which has been shown to be efficient against some Gram (-) and (+) bacteria [2], more robust and efficient molecules that can act as photosensitizers (PS) are still needed for the improvement of the technique. In this work, the second-generation PS derived from chlorophyll, photodithazine (PDZ), was submitted to a set of physiochemical variations (pH 2, 5, 7 and 10, and concentrations of 2.5, 6, 12, 16, 22, and 25 μg/mL) to promote new aggregation states with the aim of improving its antimicrobial activity. The aggregated states of PDZ were characterized by photoluminescence and photoluminescence excitation spectroscopies, showing different conformations at basic and at acidic pH, but not changing significantly with the increase in concentration. The acidic forms, which showed higher activity against both bacterial strains, were used to build composites with halloysite nanoclays, which showed a significant improvement in antimicrobial activity against S. aureus and decreased the growing rate of E. coli cells. The effect of the composites will be discussed in light of the photophysical behavior of PDZ and the near field effect.Acknowledgements:CAPES-Brazil-Finance Code 001, FAPESP 2022/08842-0.References:[1] A. O. Pereira, et al.,. ACS Appl. Mater. Interfaces 13(27), 31406-31417 (2021).[2] S. Banfi, et al., J Photochem Photobiol B 85(1), 28-38 (2006).

Photothermal properties of $Bi_2S_xSe_{1-x}$ quantum dots

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Photothermal therapy (PTT) is one of the most recent methods in the treatment of cancer, being little invasive and of low complexity. Being incipient, the production of materials with high efficiency is necessary for the technique progress. This work report the electrosynthesis of bismuth silver selenide nanocrystals ($Bi_2S_xSe_{1-x}$) stabilized by 3-mercaptopropionic acid and evaluated the performance of the material to photothermic applications. The quantum dots alloys were electrosynthed in aqueous medium by the direct reduction of Se0 and S0 in the electrochemical cavity cell.[1] The nanoalloys were obtained by adjusting the Se/S molar ratio (Se/S = 0, 0.2, 0.4, 0.6, 0.8, 1). For photothermal properties, was modulated the range of emission and photothermic response under laser excitation, λexc = 785 nm. The zeta potential mesurements indicated the negative charge associated with the particles, all most negative than −30 mV, indicating the relative stability. The samples with concentration of 61 µg/mL, when irradiated with laser 785 nm and laser potency 1 $W.cm^{-2}$, presented response between 44.4°C for 36.8°C Bi_2S_3. All chalcogenides' alloys presented increase of photoresponse, given the best photoresponse for the alloy of $Bi_2(S_{0.4}Se_{0.6})_3$, equal to 44.4°C. This behavior was associated with the structural and electronic defects, by the substitution of Se^{2-} positions by S^{2-} ions, and the intrinsic difference of orbital energies. Leading to vibrational effects generated by lattice phonons. Thus, evidence the increase of photothermal response with the chalcogenide's alloys. The proposed system represent a promisor group of nanoparticles with photoresponse, candidate to photothermal therapy of cancer.

Referências:

[1] Freitas, D. V.; Gonzales-Moya, J.R. ACS Applied Energy Materials, vol 1, 3636-3645, 2018.

[2] Yang, C.; Guo, C. ACS Applied Nano Materials, vol 1, 820-830, 2018.

Acknowledgments: CAPES, MCTI, UFPE/PGMTR, CNPq, CEMENE, CETENE

Physicochemical characterization and bioactive properties of quinoa oil (*Chenopodium quinoa* willd.) encapsulated with porcine gelatin and whey protein isolate

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The encapsulation of a lipophilic component (quinoa oil) has been used for various factors, such as the preservation and enhancement of bioactive properties, and improvement of their incorporation into a product. The aims of this work were the production and characterization of quinoa oil encapsulated in porcine gelatin (OG) and combination with whey protein isolate (OWG), using oil/water and multilayered emulsification. The characterization was performed by Dynamic Light Scattering (DLS), Fourier Transform Infrared Spectroscopy (FTIR), Zeta Potential at different pH values and the evaluation of the encapsulation efficiency. The antioxidant and antimicrobial activity of samples were performed by the methods: total antioxidant capacity (TAC), ion chelation, and minimum inhibitory concentration (MIC). The encapsulation efficiency determines the oil content (%) was successfully encapsulated at 74.13% and 83.08% for the OWG and OG, respectively. The FTIR of the OG showed an interaction between quinoa oil and gelatin indicating encapsulation of quinoa oil. OWG conferred greater protection to the oil due to interactions between porcine gelatin and whey protein, making encapsulation more efficient. The particle is 249 nm to OG and 152 nm to OWG. The zeta potential at different pHs showed that as the pH decreases, there is an increase in the zeta potential (+21 mV) considered moderately stable. The encapsulated quinoa oil OG e OWG (38,61 and 41,81 μg EAA/g), respectively, showed higher total antioxidant activity when compared to the crude oil (25,64 μg EAA/g), however in the chelation test of iron ions there was no significant difference. The MIC of *Staphylococcus aureus, Burkholderia cepacia e Burkholderia multivorans*were above10 mg. mL^{-1}. The results indicate that the encapsulation of quinoa oil shows potential for various biological applications, enhancing the activities of antioxidants e antimicrobial of quinoa oil.

Capes, CNPq

Lira K.H.D.S, et al. Plos One 15 (10) 2020.

PLGA nanoparticles functionalized with biopolymers asa new strategy for the treatment of COVID-19

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While mass vaccination against the Sars-CoV-2 virus is a promising strategy to control the COVID-19 pandemic, there is still a need to develop effective treatment options for individuals who cannot be vaccinated or for future outbreaks caused by novel Sars-CoV-2 strains. Remdesivir (RDV), a monophosphate prodrug of nucleoside analog GS-441524, was the first drug approved for the treatment of moderate to severe COVID-19. However, properties such as low solubility, high effective dose concentration, low half-life time, and side effects limit its clinical application. To circumvent this problem, we developed polymeric nanoparticles loaded with the drug RDV by nanoprecipitation methods. The nanoparticles were functionalized with dextran and chitosan to modulate cell uptake. The Remdesivir-containing nanoparticles exhibited a diameter of 120 nm, zeta potential of - 35 mV, and encapsulation efficacy of ≈ 90%. Cell internalization and cytotoxicity assays revealed that the NP@RDV significantly accumulate in different cell lines within 2 h of treatment and displayed less toxicity compared to free RDV. These results indicate that NP@RDV represents a potential new candidate for COVID-19 treatment.

Size-Dependent Synthesis and Characterization of Silica Nanoparticles: Exploring the Role of Particle Size on Material Properties

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Due to their exceptional combination of advantageous properties such as low toxicity, high biocompatibility, and biodegradability, mesoporous silica nanoparticles (MSNs) have gaineda prominent position in the scientific community for their potential applications in various fields. Silica (SiO_2) has potential applications in the biomedical field for drug delivery, tissue engineering, and diagnostic imaging, and in the environmental field for water treatment, air purification, and soil remediation. During the synthesis of MSNs, it is possible to control some parameters by adjusting the amounts of tetraethyl orthosilicate (TEOS), ammonia, andwater. It must be pointed out that the control of the morphology, particle size, and porosityof MSNs affect both the rate of release of substances and the total amount released. In thiswork, using the Stöber method, the MSNs were synthesized by hydrolysis and controlled condensation of TEOS in an alcoholic medium. The obtained nanoparticles were characterized in relation to the particle size and morphology through scanning electron microscopy (SEM), transmission electron microscopy (TEM), and dynamic light scattering (DLS) techniques. The MSNs were characterized by solid-state ^{29}Si nuclear magnetic resonance (NMR). Adsorption and kinetic studies of dye onto MSNs were also conducted. The results indicated that the MSNS was successfully obtained. Based on the results of this work, the MSNs may be used in a variety of fields, especially in hormonal monitoring of reophilic fish of the genus Brycon.

ACKNOWLEDGEMENTS

This work was partly funded by Fundação de Amparo à Pesquisa do Estado de São Paulo-FAPESP(2013/07296-2) and (2022/10016-0), CNPq (309711/2019-3) and CAPES (88887.641038/2021-00), (88887.643328/2021-00) and (8887.828064/2023-00).

Studying the interaction of octaketide cytosporone-bwith Langmuir thin films as cell membrane models

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The study of compounds with biological action on cell membrane models enables us to predict the interactions at the molecular level. Such interactions can be investigated using Langmuir thin films of phospholipids, which are monolayers assembled at the air-water interface that can mimic one side of the cell membrane. This work aims to study the interaction of Cytosporone-B (Csn-B), a secondary metabolite isolated from the fungus Phomopsis sp., with Langmuir monolayers composed of the lipids dipalmitoyl phosphatidylcholine (DPPC), palmitoyloleoyl phosphatidylcholine (POPC), dipalmitoyl phosphatidylethanolamine (DPPE), dioleoyl phosphatidylethanolamine (DOPE), dipalmitoyl phosphatidylserine (DPPS) and palmitoyloleoyl phophatidylserine (POPS), major lipids found in erythrocyte cell walls (both choline phospholipids), in bacteria cell walls (both ethanolamine phospholipids), and in the cell membranes in the early stages of apoptosis (both serine phospholipids), respectively. Csn-B shifted the isotherms to lower areas, pointing to monolayer condensation, which suggests that the Csn-B interacted with the polar head of the phospholipids, reducing their lateral repulsion. AFM images showed that the different monolayer compositions adhered in different ways to the substrate after the addition of Csn-B. BAM images showed the formation of domains and discrete clusters after the addition of Csn-B. PM-IRRAS spectra showed changes in the relative intensity of bands related to the stretching of the methyl group present in the large alkyl chains of the lipids, as well as in the phosphate and carbonyl group bands, indicating not only interaction with the polar head but also the distortion of tilting of the alkyl chains. In conclusion, Csn-B changed the thermodynamic and morphological properties of lipid monolayers, where it is possible to notice that the film''s chemical composition modulates the interaction with cell membrane models.

Acknowledgments: CAPES, FAPESP, and CNPq.

Study of Encapsulation of Antineoplastic Drugs in Nanostructured Systems: Structural Characterizationand Synergistic Effect of Drugs

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The nanotechnology field has been growing in the past decades, improving studies of several compounds. Drug delivery systems are being largely used, whose objective is to increase pharmaceutical substances efficacy, and this may include a more selective distribution within the sick organism, the molecules liberation time fine-tuning and/or attenuation of adverse effects. Drugs are nanoencapsulated within synthetic origin or biodegradable structures, necessarily compatible with the active principle. Cubosomes are a hotspot, which are complex nanostructures that successfully encapsulate hydrophilic and hydrophobic activities. The main objective is to study the effect of the antineoplasic activity against cervical cancer cells (HeLa) and human epidermal keratinocyte (HaCat), the synergic/antagonistic effects of drugs, cubosomes synthesis and characterization for further encapsulation of actives inside them. The drugs are cisplatin, vemurafenib and curcumin. In this work, we will broaden our knowledge of the antineoplasic activity of these compounds, testing them on HeLa and HaCat cells, and quantifying the post-exposure cell viability. The cubosomes were synthed with phytantriol and tested by dynamic light scattering (DLS), nanoparticle tracking analysis (NTA) and cell viability test. The cell viability test showed promising results at a dosage of 0,01 mg/mL in both cell lineages. The cubosomes had a hydrodynamic diameter of 197(5) nm, polydisperse index of 0,05(0,05) and a concentration of 5,2 x 10^{12}particles/mL. The encapsulation looks promising and the cubosomes were efficiently synthed and seem to be stable.

Acknowledgments:
FAPESP, CAPES and CNPQ

References:

[1] B. MALHEIROS,R. D. CASTRO, M. C.G. LOTIERZO, B. R. CASADEI, L. R. S. BARBOSA. Design and manufacturing of monodisperse and malleable phytantriol-based cubosomes for drug delivery applications. Journal of Drug Delivery Science and Technologies, Switzerland, v. 61, n. 1, p. 1-8, 2021.

SYNTHESIS AND CHARACTERIZATION OF NICKELAND COPPER DOPED COBALT FERRITE NANOPARTICLES FOR DRUG DELIVERY

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The properties of Magnetic nanoparticles (MNPs) MNPs allow the absorption, transport and controlled delivery of drugs to a specific body-place with high efficiency by means of changing the intensity of a magnetic field [1]. Among MNPs, cobalt ferrite (CoFe2O4) is a promising material for drug delivery, due to its physical-chemical and mainly magnetic properties [2]. Thus, in the present work, samples of CoFe2O4 doped with copper and nickel were obtained from two synthesis routes: coprecipitation and sol-gel, using different percentages of doping and subsequently thermally treated at temperatures of 700, 800 and 900 °C. The formation of a single CoFe2O4 phase with a cubic structure was observed in all samples treated in the range of 700 to 900 °C. The magnetic behavior of the samples was analyzed through VSM. The infrared spectra indicate the presence of modes associated with cobalt ferrite. One can verify the active modes of the spinel structure through the Raman spectra. Based on the achieved results, it is concluded that the synthesis routes were efficient for producting cobalt ferrite NPs, which can be used later in the delivery of drugs.

Keywords: Cobalt ferrite, Magnetic nanoparticles, Delivery of medicines.

Acknowledgments: CNPq, Capes, FAPEPI and FAPEMA.

References

[1] TAMBOLI, Qudsiya Y. et al. Green Synthesis of Cobalt Ferrite Nanoparticles: An Emerging Material for Environmental and Biomedical Applications. Journal of Nanomaterials, v. 2023 (2023).

[2] GHANBARI, Meysam; DAVAR, Fatemeh; SHALAN, Ahmed Esmail. Effect of rosemary extract on the microstructure, phase evolution, and magnetic behavior of cobalt ferrite nanoparticles and its application on anti-cancer drug delivery. Ceramics International, v. 47,n. 7, p. 9409-9417 (2021).

Synthesis and characterization of SPIONs functionalized with Cysteine and Glutathione forbiomedical applications

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Motivated by the broad range of applications in the technological, industrial, and biomedical fields, we developed mechanisms for synthesizing superparamagnetic iron oxide nanoparticles (SPIONs) for biomedical use [1,2]. In this research, we synthesized, characterized, and evaluated the cytotoxicity of this nanomaterial, comparing and investigating its characteristics with the available literature to improve its biocompatibility and suitability for nanomedicine applications.Synthesis is critical in obtaining the necessary properties for the objective application of nanomaterials. Among the various existing methods, we selected co-precipitation because it is a simple and cost-effective method that produces nanomaterials compatible with biomedicine [3]. We synthesized SPIONs by mixing ferric and ferrous chlorides in an aqueous solution and precipitated them using ammonium hydroxide. To prevent agglomeration of nanoparticles and facilitate targeting of specific sites using an external magnetic field, we functionalized the SPION surface with organic ligands (Cysteine or Glutathione - molar ratio, SPIONs:ligand = 1:5) that contain a functional group (-SH).We characterized the particles using Fourier transform infrared spectroscopy (FTIR), UV-Vis spectroscopy, Microscopy RAMAN, X-ray powder diffraction (XRPD), dynamic light scattering (DLS), and SQUID magnetic measurements. Finally, we evaluated the cytotoxicity of nanoparticles in vitro using healthy and tumor cell cultures.

Acknowledgments: We would like to acknowledge the support provided by FAPESP (2022/03647-4), CAPES and the Multiuser Experimental Centre (CEM) from UFABC and CESM - UNIFESP.

References:[1] Campos, I.; et al. Sn Applied Sciences, v. 2, 2020.[2] Martins, E. S.; et al. Chemistryselect, v. 6, p. 7931-7935, 2021. [3] Britos, T. N.; et al. Materials Science Engineering C-Materials for Biological Applications, p. 171-179, 2019.

Synthesis and investigation of new crystalline forms of antineoplastic agents

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Cancer is one of the most complex public health problems in the Brazilian health system. In 2020, more than 19 million cancer cases were diagnosed. Melphalan hydrochloride (MEL) is a chemotherapeutic drug with antitumor activity known for its groups of alkyl agents. During the last decades, MEL has been administrated to patients with breast and ovarian cancer, mainly to treat multiple myeloma. However, its oral administration has some limitations as pre-systemic elimination and incomplete toxicity [1]. Herein, we determine the crystal structure of MEL hydrochloride using X-ray powder diffraction data and a simulated annealing procedure. MEL crystallizes in a monoclinic crystal system ($P2_1$). A rational screening conducted in Mercury (CSD-Materials) indicates the possibility of using different synthons to obtain new crystalline forms to improve the drug' s performance and the compound' s-controlled release. As MEL displays low water solubility, new co-crystal formsare of interest. The milling process is performed using mortar and pestle (manual milling)and ball mill (mechanochemical). In both procedures, stoichiometric mixtures of MEL are combined with some conformers (L-glutamic acid, Lactose, Saccharin, and Caffeine), which Mercury indicates as possible synthons.

ACKNOWLEDGMENTS

The authors thank the financial support provided by CNPq (305601/2019-9), FAPESP (2021/03640-7), CAPES Finance code 001, and the Multiuser Experimental Center (CEM-UFABC) for experimental support.

REFERENCES

[1] RAJPOOT, P.; BALI, V.; PATHAK, K. Anticancer efficacy, tissue distribution and blood pharmacokinetics of surface modified nanocarrier containing melphalan. Int. J. Pharm. (2012) 426, 219-230.

Synthesis, Characterization, and in vitro Cytotoxicity of Gemcitabine-loaded Luminescent Calcium Orthophosphate Nanoparticles

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Pancreatic cancer occupies the fourth position in deaths caused by malignant neoplastic diseases and exhibits high metastasis progression rates [1]. In modern nanomedicine, theranostic nanocarriers are in the front-line search for innovative solutions to improve the efficiency of traditional therapeutic methods, such as chemotherapy. One of the most promising theranostic nanosytems is based on calcium orthophosphate (CaP) nanoparticles (NPs), which exhibit high biocompatibility, loading efficiency of drugs, and self-activated luminescence [2,3]. This study aims to synthesize luminescent CaP NPs and use them for the controlled delivery of gemcitabine (GEM), which is one first line therapeutic agent often administered to treat pancreatic cancer. The CaP phase and the presence of GEM on the NPs were confirmed via FTIR spectroscopy. The typical broadband photoluminescence (PL) emission of CaP centered at 580 nm (λexc = 405 and 488 nm) was observed via PL spectroscopy. Size of NPs was estimed as 206.1±19.37 nm and 206.3±68.0 nm via DLS andNTA techniques, respectively. The Zeta potential decreased from −1.88 ± 1.21 mV to −13.0 ± 0.153 mV due to GEM encapsulation. The GEM loading efficiency of 9.8% was determined by UV-Vis spectroscopy. The cell viability of our NPs was probed via MTT assay with PANC-1 cell line, showing statistically significant reduction at concentrations ≥ 40 ug/mL and 10 ug/mL of GEM-CaP NPs in 48 and 72h of incubation, respectively, and IC50 at 160 ug/mL and 40 ug/mL of NPs in 48 and 72h of incubation, respectively. The present results are promising for future procedures in nanomedicine using GEM-CaP NPs.

Acknowledgments:
Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP), grants nº 2020/14417-4and 2021/11424-2, and CNPq, grant nº 164373/2020-9.

References:
[1] J.D. Mizrahi et al., Lancet, 2020, 395, 2008-2020.
[2] C. Qi, et al., Chem. Soc. Rev., 47, 357-403, 2018.
[3] T.R. Machado et al., Mater Today Chem, 14, 100211, 2019.

SYNTHESIS OF PEI-*b*-PEGMA-*b*-PCL COPOLYMER MICELLES AIMING FOR CONTROLLED DRUG RELEASE APPLICATION

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Oral biofilms are structures that shelter different types of microorganisms. Biofilms are formed when planktonic microorganisms attach to teeth surface and buccal mucosa and start secreting an extracellular polymeric substance (EPS), composed of extracellular nucleic acids, proteins, and exopolysaccharides, which gives the structure a net negative charge. Oral biofilms can cause periodontal disease, endodontic infections, and dental caries, and are also related to systemic deceases, like cardiovascular disease, rheumatoid arthritis, and respiratory disease, according to some evidence. The biofilm matrix, consisting of EPS, has channels for transporting nutrients and water and protects the residents against the outer environment, immune system attacks, and antibiotic substances [1]. Therefore, the goal of this work is the synthesis and physicochemical characterization of polymeric micelles of the tri-block PEI-*b*-PEGMA-*b*-PCL aiming for the application in the treatment of oral biofilms. The polymer is synthesized in two stages, firstly via ring-opening polymerization of ε-caprolactone with PEGMA [2] as initiator and Tin (II) 2-ethylhexanoate ($Sn(Oct)_2$) as the catalyst and then functionalized with PEI by aza-Michael addition in mild conditions. 1H NMR and FTIR spectroscopies confirmed the formation as PEGMA-*b*-PCL, which presented an average ζ-potential 7.58 mV in a pH 4. The addition of PEI is expected to raise the positive surface charge, enabling greater electrostatic interaction between the nanoplatform and the biofilm.

Acknowledgments: CNPq, LaMMC, IQ UNESP.

References:

[1] Panariello, B. H. D., de Araújo Costa, C. A. G., Pavarina, A. C., Santiago, S. L. Duarte, S. Advances and Challenges in Oral Biofilm Control. Curr Oral Health Rep 4, 29-33 (2017).

[2] Brandt, J. V. et al. Synthesis and colloidal characterization of folic acid-modified PEG-b-PCL Micelles for methotrexate delivery. Colloids and Surfaces B: Biointerfaces 177, 228-234 (2019).

Synthesis, standardization and characterization of silver nanoparticles with red propolis from Alagoas

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The main antimicrobial assets of today are silver nanoparticles (NPAg). Several methods ofsynthesis of silver nanoparticles are used, among them, biogenic synthesis, which consists ofthe use of biological materials as a reducing/stabilizing agent. The Alagoas red propolis(RPA), has several biological activities, such as: antibacterial, antifungal, antioxidant andanti-inflammatory, showing a promising biological agent in the synthesis of silvernanoparticles. Silver nanoparticles with red propolis from Alagoas (NPAg-RPA) wereobtained through biogenic synthesis and characterized by UV-Vis, DLS, Fluorimetry, PXRD,FT-IR and Raman techniques. By UV-Vis was observed band around 414nm, referring to theSPR band, inducing the formation of nanoparticles. Through the PXRD, peaks at angles 2θwere observed in 38.04, 44.80 and 64.54, which refer to the crystalline planes of metallicsilver. By the FTIR technique was observed an interaction of silver with red propolis, thedisplacements of bands observed indicate the participation of RPA in the reduction of silverand formation of NPAg-RPA. By Raman spectroscopy, bands close to carbonyl vibrations areobserved, indicating the participation of RPA in the stabilization of NPAg-RPA. The analysisof fluorimetry indicated a decrease in the fluorescence intensity of silver nanoparticle after theformation of NPAg-RPA, proving its achievement. The compound obtained in the presentwork represents a promising nanobiotechnological compound with antibacterial activity,especially for topical applications.

[1] ALENCAR, S. M. et al. Journal of Ethnopharmacology, v. 113, p. 278-283, 2007.
[2] GHRAMH, H. A. et al. Science of Advanced Materials, v. 11, p. 1-8, 2019.
[3] ZHANG, X. F. et al. International Journal of Molecular Sciences, v. 17, n.9, p.1534, 2016.

Tailoring the intrinsic luminescence in hydroxyapatite nanoparticles via defects manipulation

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Hydroxyapatite (HA) is one of the most studied phase of the calcium orthophosphate family of compounds due to its excellent properties of biocompatibility, bioactivity, and controlled biodegradability [1]. HA nanoparticles (NPs) exhibit self-activated photoluminescence (PL) under UV-Vis irradiation, which can be used for distinct applications such as cellular imaging for the diagnosis of diseases including various types of cancer [2]. The PL property in HA NPs is mainly caused by the presence of a large density of point and structural defects, which can be tuned to modify the main characteristics of the emission band, such as intensity, emission color, lifetime and quantum yield [3]. This study aims to demonstrate the influence of carbon impurities on the luminescence of HA NPs synthesized by chemical precipitation followed by heat treatment at 400ºC/4h. Samples were initially characterizedvia X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Thermogravimetric analysis (TGA), and transmission electron microscopy (TEM). Then, PL spectroscopy revealed the impurities have profound impact on PL properties of HA including the intensity, color, and broadness of the emission band. Then, the cell viability of our HA NPs was probed via MTT assay, and their performance in cell imaging was tested via confocal microscopy, showing promising results for future bioimaging procedures in nanomedicine.

Acknowledgments:

Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP), grants nº 2022/14435-8, 2020/14417-4, and CNPq, grant nº 164373/2020-9

References:

[1] C. Qi et al., Chem. Soc. Rev., 47, 357-403, 2018.

[2] T.R. Machado et al., Advances in the Synthesis and Applications of Self-Activated Fluorescent Nano- and Micro-Hydroxyapatite. Springer, Cham, 2022.

[3] T.R. Machado et al., Mater Today Chem, 14, 100211, 2019.

The effect of pesticide cocktail in the bioinspiredternary lipid monolayer

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Pesticides are chemicals used to protect the crops against intruders that can damage the plantation. However, their irregular use can lead to environmental contamination and, consequently, human intoxication. Furthermore, the mixture of two or more contaminants can favor the "cocktail effect", i.e., the combined effect of multiple products in the human health. Acephate is an insecticide from the organophosphorus family, can cause disorders in the human neurological system, and was the first pesticide found irregularly in Brazilian crops between 2017 and 2018. Diuron is an urea-class herbicide mainly used in sugarcane plantations and has carcinogenic potential in the bladder region. In order to investigate the combined effect of the acephate/diuron mixture on the bioinspired cell membrane model, Langmuir monolayers of POPC, cholesterol (Chol) and sphingomyelin (SM), and their mixture (ternary monolayer - 70/20/10 mol%, respectively) were explored in this study. The Langmuir monolayers were characterized by π-A isotherms and PM-IRRAS (Polarization Modulation-Infrared Reflection Adsorption Spectroscopy) in absence and presence of the acephate/diuron cocktail. A shift to higher molecular areas was observed to the π-A isotherms of POPC and ternary monolayers, while for Chol and SM no significant changes were noticed. A more detailed investigation was reached at intermolecular interaction by using PM-IRRAS, revealing that the pesticide mixture affects the polar head group of POPC, which is in agreement with molecular dynamics simulation. Acephate/diuron also affect the polarhead group of SM lipids and, additionally, the amide-I region. The intermolecular interaction between the pesticides with POPC and SM were also observed in the ternary lipid monolayer. This study finds evidences in the harmful effects of acephate and diuron in the lipid assembly that maintains the integrity of cell membranes.

Acknowledgment: CAPES, INEO, FAPESP, CNPq

Theranostic nanoparticles based on dual therapy: Proton Sponge and Nanorods against tumor normoxia and hypoxia conditions

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Tumor hypoxia is increasingly recognized as an important detrimental factor in cancer therapy, compromising therapy and driving malignant progression [1]. Conventional chemotherapy is increasingly suffering from drug resistance in advanced stages of cancer, leading to trials and the use of new therapies [2,3]. Our study consisted in the application ofa dual therapy based on thermal and tamponade therapy, in the battle against pancreas (PANC-1) and lung (A549) cancerous cells. Traditional MCM41 and Periodic mesoporous organosilicas (PMOs) sensitive to pH were synthed as a shell around gold nanorods through the sol-gel method, obtaining stable nanoparticles of 180 - 230 nm with hexagonal pore ordering. A model drug for studying the buffer effect was TMGN (1,8(tetramethylguanidino)naphthalene), known as proton sponge, in which the encapsulation in the pores of the silica NP was about 30%. Photothermal experiments showed that despite the silica shell, the nanorods exhibited a temperature rise of 15°C after10 minutes of illumination with an 808 nm laser. Cell viability studies showed that the nanoparticles, when incorporated individually (laser off), had greater effects on hypoxic cells than those incubated under normoxia, in addition to the fact that MCM-41 has no toxicity. The toxic effect without laser application at 808 nm (hyperthermia) was associated with the proton sponge action. When the laser was activated, we observed a synergistic effect between the therapies (buffer and hyperthermia) in both cell lines, as well as an increase in drug delivery in the presence of the high temperature, leading to a new theranostic platform for anti-cancer application.

Acknowledgment: CAPES, CNPq and FAPESP (Grant number 17/22056-9)

[1] Qin, B. et al. J Nanobiotechnol 19, 427 (2021).
[2] Housman G., et al. Cancers (Basel). 2014 Sep 5;6(3):1769-92.
[3] Crezee J., et al. Cancers (Basel). 2021 Mar 12;13(6):1240

Toxicological evaluation of Calcium and EuropiumDoped Titanium Dioxide Nanocrystals

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Titanium dioxide (TiO2) nanocrystals (NCs) have wide industrial and pharmaceutical application. Due to its ability to absorb UV light and high refractive index, TiO2 is used in sunscreen, and in paints and foods as dyes. The applicability of TiO2 raises questions about its safety, both for the environment and for human health, since evidence indicates that TiO2 has cytotoxic, genotoxic effects and carcinogenic potential. Therefore, studies that determine the toxic effects of nanomaterials and methodologies, such as doping, that bring new properties to nanomaterials are essential. Thus, this work aimed to evaluate the toxicological effects of TiO2 NCs doped with Europium and 0.5 Calcium (TiO2:4Eu:05Ca) in *Drosophila melanogaster.*30 first instar larvae (n=6) were orally exposed to different concentrations of TiO2:4Eu:05Ca (0.5; 1.0; 2.0; 4.0 mg/ml) added to standard medium, and the effects during the development of the animals were analyzed. The larval and pupal lethality rates, the development time and the lifespan of the animals were quantified and showed no differences when compared to controls. Damage to the intestinal epithelium was measured using the trypan blue exclusion method. The results indicate that the NCs were biocompatible at the tested concentrations. However, further studies are needed to fully understand the mechanisms by which Calcium improved TiO2:4Eu:05Ca biocompatibility.

Understanding the interaction of acetogenin withcancer cell membrane models by DPPS Langmuirmonolayers

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Acetogenins are a class of biomolecules derived from Annonaceae family plants which recently has shown cytotoxic properties, along with antitumor and antiparasitic activity. However, as its biological activities are widely studied, its molecular mechanism of action is not yet known. As a result, it is crucial to understand if the drug acts by provoking alterations in the physical-chemical properties of the cell membranes or if it penetrates the membrane and acts on intracellular structures. It is also relevant to study the interaction of such kind of bioactive molecules with phospholipids to develop possible drug carrier systems, such as liposomes. To fill these gaps, we studied the action of an unsaturated acetogenin named PMD3, extracted from Porcelia macrocarpa seeds, in cell membrane models at the air-water interface by Langmuir monolayers composed of the phospholipid dipalmitoylglycerophosphoserine (DPPS), since phosphatidylserines, predominant lipids in the inner leaflet of the plasma membrane, are encountered at high concentrations in the outer leaflet of tumor cell membranes. The characterizations of pure DPPS and mixed (DPPS + PMD3) films were made by surface pressure-area isotherms, hysteresis and tensiometric stability assays, polarization modulation infrared reflection absorption spectroscopy (PM-IRRAS) and Brewster angle microscopy (BAM). The introduction of the drug on the lipid monolayer caused a higher orientational disorder, lower stability, and other alterations in the physical-chemical properties of the film. PM-IRRAS spectra show that PMD3 interacts on both the alkylic and polar portions of DPPS. These results may be related to the mechanism of PMD3 on tumor cell membranes and should be fundamental to better selecting tumorigenic drugs as bioactive materials.

Unveiling the Ultrastructure of Microbial Cell Walls: Investigating the Effects of AIP6 Cell-Penetrating Peptide Using Atomic Force Microscopy

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The pentapeptide RLRWR (AIP6) belongs to the group of cell-penetrating peptides (CPPs), being one of the shortest sequences capable of translocating biomembranes. While the interaction of CPPs with mammalian cells has been extensively investigated, studies on the effects of these molecules on the cell wall of microorganisms have not been carried out at the same intensity. Herein, we investigated the interaction of the AIP6 peptide with the cell wall of model microorganisms using Atomic Force Microscopy (AFM) techniques capable of providing ultra-high resolution and detailed physicochemical data. We developed analysis protocols that allowed us to obtain three-dimensional details of these interfaces, both in fixed samples and living cells maintained under hydrated conditions. We also characterizedthe spectroscopic signature of the cell wall at the nanoscale. The model organisms investigated in this study were the procaryotic E. coli and B. subtilis, whereas the eukaryotic model was the yeast S. cerevisae. Topography AFM data and nano-spectroscopy (AFM-IR) experiments were performed on PFA-fixed slides containing cells previously incubated with AIP6. Live cell imaging was conducted using BioAFM with culture medium-containing cells adhered to polylysine-treated glass slides. MICs were found in the range of 0.1-1mg/ml, and AIP6 was found to be selective to the Gram-positive B. subtilis. Ultrastructural analysis of the cell wall allowed the observation of pores, and the formation of peptide domains was identified through the spectroscopic signature. The AIP6 peptide showed affinity for the cell walls of bacteria, especially B. subtilis, while no action was observed against S. cerevisiae. In prokaryotes, the formation of peptide domains that likely induce the formation of pores responsible for cell lysis was observed.

Acknowledgements: To FAPESP (grant n° #2022/03056-6) and CNPq (409455/2018-0) for financial support. To CAPES (finance code 001) for fellowships.

Water contaminants combined effects on lipid monolayers and bilayers as cell membrane models

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The investigation of the impacts of emerging pollutants on cellular systems is crucial for comprehending their potential risks to human health and the environment and developing effective strategies for their mitigation. Some emerging pollutants, such as BPA, some pesticides, and hormones have some of their effects known. However, the toxicity of emerging pollutants in mixtures can generate effects that are different from those observed when each individual compound is evaluated separately. These combined effects of water contaminant mixtures may be additive, synergistic, or agonistic. This work discusses the issue of emerging pollutants and their potentially harmful effects on human health and the environment, with a particular focus on two types of pollutants: antibiotics (amoxicillin) and dyes (methylene blue). The study examines the effects of these pollutants and their mixture on cell membrane models based on lipid monolayers and bilayers. Using Langmuir films, the study revealed a synergistic effect for the mixture of pollutants on lipid monolayers through π-A isotherms and PM-IRRAS spectroscopy. In contrast, the bilayer systems from giant unilamellar vesicles (GUVs) and contrast phase microscopy experiments suggested similar molecular effects for individual pollutants and the mixture, with a predominance of the MB effects. The study considered the critical factors of chemical interactions and molecule for their different effects on the cell models. This research emphas the importance of comprehending the molecular impacts of emerging pollutants on human health and developing models to investigate their interactions with the cell membrane.

Acknowledgments: CNPq, FAPESP, and CAPES.

Zein-propolis Electrospun nanofibers: a potent antimelanoma material

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Propolis is a natural resinous substance produced by bees, which have many biomedical properties, such as antitumor and immunomodulatory activity[1]. The main problem using it as a therapeutical agent is its high hydrophobicity. Using zein, the main protein in corn with over 50% of its amino acids present an apolar character easing hydrophobic interactions[2], to electrospun nanofiber formation, propolis can be controlled released in biological environment. Considering this, we prepared thermally annealed zein-Propolis electrospun nanofibers with no or 45mg/mL of propolis, that already showed no cytotoxic effects in non-tumoral skin cell line [3]. After nanofiber preparation, human melanoma and melanocyte cell lines (A375 and NGM cell lines, respectively) were plated over nanofibers. Cells were allowed to interact with nanofibers for 72 h. After, cell viability was measured by neutral red uptake. Results showed reduction in melanoma cell viability and increase of neutral red uptake from melanocyte cell line, indicating that the nanofiber can be used as a delivery platform for propolis to cells and that melanoma cell viability are more affected than non-tumoral cells. Further experiments need to be performed using other cell lines to determinate this specific activity, and evaluating why there is difference between the tumoral and non-tumoral cell lines. In conclusion, thermally annealed zein-Propolis electrospun nanofibers, a natural biocompatible material, appears to be a potent material with specific cytotoxic activity on human melanoma cells while enhancing non-tumor cell proliferation.

Acknowledgements:
CAPES(PROAP-PPGBCM), PRPPG-UFPR(Edital apoio a pesquisa), CAPES and CNPq student’s fellowship.

References:

[1] W, HUO et al., J Cereal Sci., 79, 354-361 (2018); [2] T, ALBERTI et al., Nanotechnol Adv Mater Sci, 1, 1-10 (2019); [3] S, HOCHHEIM et al., Macromol. Biosci. Online ahead of print (2023).

G-Photocatalytic materials applied in energy conversion and environmental remediation

How Photocatalytic Materials can contribute for a Sustainable Agriculture

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The literature about application of photocatalytic materials is continuously introducing new promising catalysts, but the context of applications is still far from consistent scenarios, based on actual demands. In particular, agriculture can be benefited by these technologies if they can attend specific challenges, which frequently requires robustness and adequacy to be applied to extremely high scales. Thus, the understanding of the boundary conditions can helps to the development of feasible solutions based in the current knowledge. Here we propose to revise several applications, from the literature and of our research group, where photocatalytic materials are playing a role – as CO2 reduction, CH4 reforming, NO3 and N2o oxidation, among others. A revision about the efforts on the more consolidated applications (e.g. photdegradation of agrichrmicals in water) to those still in infancy (e.g. N2 reduction) can give a good portfolio for materials scientists to contribute to a sustainable agriculture.

Misfit Ca-cobaltites as promising electrodes for fuelcells and electrolyzers

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This lecture focuses on the review the recent efforts of the Materials and Electrochemistry Interdisciplinary Group of the Federal University of Paraíba (GIME-UFPB) on the development of misfit calcium cobaltites (Ca3Co4O9, C349). These materials have emerged as promising functional electrodes for oxygen reduction and oxygen evolution reactions. The effects of Cu-doping and PrOx addition on the electrochemical activity of C349-based electrodes have been evaluated under typical operational conditions of solid oxide fuel cells (SOFCs) and alkaline electrolyzers. Powders obtained by a proteic sol-gel method and by solid state reaction, using mollusk shell as a natural source of calcium carbonate, have been systematically studied using a variety of characterization techniques. Electrodes were electrochemically characterized by impedance spectroscopy as a function of the temperature and oxgyen partial pressure. The electrochemical assessment indicated the materials developed at GIME-UFPB are very competitive in comparison to those from literature, highlighting the high potential of Ca-cobaltites for energy conversion applications.

Acknowledgements:
The author acknowledges CNPq and CAPES for their financial support.

References:

[1] Erik B.G.A. Fulgêncio, Klívia P.V. Melo, Rinaldo M. Silva, João P.F. Grilo, Vincent Caignaert, Moisés R. Cesário, Liszandra F.A. Campos, Daniel A. Macedo, Ceramics International, Volume 43, 9564-9567 (2017).
[2] Erik B.G.A. Fulgêncio, Francisco J.A. Loureiro, Klívia P.V. Melo, Rinaldo M. Silva, Duncan P. Fagg, Liszandra F.A. Campos, Daniel A. Macedo, Journal of Alloys andCompounds, Volume 788, Pages 148-154 (2019).
[3] Thayse Silva, Vinícius Silva, Jakeline Santos, Thiago Simões, Daniel Macedo, Materials Letters, Volume 298, 130026 (2021).

Photocatalytic Reduction of CO2 using hybrid carbon-based materials

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Replacing metallic catalysts with metal-free abundant materials has become a priority to boost sustainable and renewable solutions towards current energetic and environmental challenges. Many different forms of carbon have shown high catalytic activities, with better performance and durability than certain state-of-the-art non-precious metal catalysts for energy conversion/storage and environmental remediation. Among them, nanoporous carbons have attracted the attention since the outstanding role of nanopore confinement has led to reporting high photochemical quantum yields in a high number of oxidation and reduction reactions due to light activation of optically active sites. The stabilization of the photogenerated excitons in the nanopores facilitates molecular transformations of interest, such as the conversion of CO_2into fuels and added-value chemicals. In this study we have explored the use of nanoporous carbons with controlled pore architectures as photoelectrocatalysts for the reduction of CO_2, using supported electrodes (e.g., glassy carbon, titanium foil) and a cell provided with a quartz window and various irradiation sources (simulated solar light, LEDs). Some carbon materials showed a high conversion of CO_2into CO with a high selectivity for CO over H_2. Such selectivity correlates well with the electrochemically active surface area of the materials and it was higher for selected carbons doped with heteroatoms, due to the presence of surface defects upon functionalization. The studied carbons also displayed a good stability after several hours of reaction, as seen in the post-mortem analysis of the catalysts. We herein provide a comprehensive interpretation of the origin, reactivity and stabilization pathways of the charge carriers, with perspectives of utilization in photoelectrocatalytic reactors.

Acknowledgements.The financial support from the European Research Council (grant 648161) and Région Centre Val de Loire (grant MATHYFON) is acknowledged.

Shining a Light on Sustainable Chemistry: Harnessingthe Power of Photoelectrocatalysis for Clean Energy Conversion and Environmental Remediation

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Photoelectrocatalysis has emerged as a promising approach to address global challenges in energy conversion and environmental remediation, owing to its ability to simultaneously harness solar energy, facilitate charge transfer, and catalyse chemical reactions. The use of light as an energy source and electron transfer mediator enables the development of efficient and selective reactions that can convert solar energy into useful fuels or degrade harmful pollutants. The ability to convert solar energy into clean fuels, such as hydrogen, has the potential to transform the energy landscape and reduce dependence on fossil fuels. Furthermore, the use of photoelectrocatalysis for environmental remediation, such as the degradation of organic pollutants or nitrogen fixation for fertilizer production, can contribute to mitigating climate change and improving human health.

In this lecture, I will explore our research into earth-abundant and low-cost metal oxides for hydrogen evolution, biomass conversion, and air pollutants (such as NOx) abatement [1]; as well as carbon-based nanomaterials for de-halogenation and organics conversion [2]. For all these materials, I will show how combinations of electrochemical and magnetic resonance techniques afford a better understanding of the underlying mechanisms of photoelectrocatalytic processes, opening up exciting new avenues in sustainable chemistry for energy and the environment.

By continuously pushing the frontiers of what is achievable by converting solar energy efficiently, photoelectrocatalysis exemplifies the importance of interdisciplinary collaborations and creative thinking in tackling some of the world''s most pressing challenges.

References:
[1] Phys. Chem. Chem. Phys. 2017, 19, 32678-32686; ACS Catal., 2018, 8, 6927-6938; RSC Adv., 2018, 8, 27674-27685.
[2] Chem. Eur. J., 2018, 24, 4382-4389; Adv. Synth. Catal., 2021, 363, 4740-4753; Angew. Chem. Int. Ed., 2020, 59, 4106-411.

Application of carbon fiber (CF/WO_3) used as photoactivated filtration membrane for sertraline removal

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The application of photoactivated filtration membranes results in a promising alternative in the treatment of contaminated water since they allow the simultaneous retention/removal of emerging contaminants and minimize problems associated with filtration such as incrustation, deformation, continuous maintenance and less reusability. The present work proposes the hybridization of the filtration/photocatalysis systems, from the use of carbon fibers (CF) modified with n-type semiconductor (WO_3), via electrodeposition and heat treatment at 450 °C/2h, applied in the removal of Sertraline (SER); an antidepressant of extended use and with potential contaminant of water environments. Thermogravimetric analysis showed decomposition of C above 750 °C, proving the stability of CF for its modification by heat treatment. The micrographs showed uniform WO_3 deposition on the CF surface. The X-ray diffraction patterns showed characteristic peaks for (C and WO_3) and the presence of the monoclinic phase (high photoactive). The XPS analysis showed an asymmetrical peak of greater intensity at 530.3 eV, attributed to metallic bonding (W-O), and the presence of double symmetrical peaks at 35.3, 37.6 eV and 41.3 eV attributed to levels W $4f_{7/2}$, W $4f_{5/2}$ and W $5p_{3/2}$. The modified CF showed broad UV-Vis absorption (300 to 700 nm) and bandgap energy (E_g) 3.2 eV, as well as an increase in BET area from 0.2045 ± 0.0091 to 3.0826 ± 0.0224 m^2/g after WO_3 deposition with pore size between 13 and 33 nm characteristic of mesoporous materials. The filtration membranes showed high efficiency reaching complete removal of SER [1 mg L^{-1}] in 90 and 30 min when evaluated in Milli-Q water and in real effluent, as well as 66% degradation after 5 reuse cycles. The use of scavenger agents determined the fundamental role of hydroxyl radicals ($HO^{\cdot}$) and superoxide ($O_2^{\cdot-}$) in the SER degradation mechanism.

The authors thanks, Brasil: INCT-DATREM, FAPESP, CNPq, CAPES and Chile: CONICYT, FONDEQUIP, DICYT USACH.

Bactericidal and virucidal activity of polymer-basedcopper coating boosted by visible light radiation

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Before the pandemic caused by the severe acute respiratory syndrome coronavirus 2 which caused the death of more than 6.5 million people worldwide, the research of virucidal materials has been encouraged [1]. However, besides viruses, bacteria also represent a threat to human health as they are commonly responsible for nosocomial infections that exhibit a significant mortality rate. Among the materials studied, copper stands out due to its intrinsic biocidal properties [2]. The present work developed composite films containing different copper particles into a polyvinyl alcohol matrix. By applying the films with copper micro- and nanoparticles, *Staphylococcus aureus*population was reduced above 6 log orders after 4 h of incubation in the absence of light and under light illumination. The films with copper(I) oxide presented the same result only under visible radiation, proving that under illumination the material induces a quicker cell death. *Escherichia coli*showed more resistance, since in 4 h only the films with copper nanoparticles presented bactericidal activity. The films containing copper microparticles were tested against murine hepatitis virus, and 99.99% of the virus was inactivated after 30 min of contact. Thus, the composite films are an encourage strategy to combat any infection that can be contracted by common touching surfaces.

Acknowledgements:

This work was financially possible due to Human Resources in Strategic Areas (CNPq #400912/2019-8) and Plasmar Tecnologia Ltda.

References:

[1] JANA, I. *et al.* ACS Applied Nano Materials 4, p. 352-362 (2021).

[2] MCEVOY, J. G.; ZHANG, Z. Journal of Photochemistry and Photobiology C: Photochemistry Reviews 19, p. 62-75 (2014).

Bactericidal photocatalyst of TiO_2-based copper under visible light radiation

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In recent decades, there has been growing interest in developing photocatalytic materials to address environmental and health-related issues. Following the SARS-CoV-2 pandemic, many researchers have focused on developing materials with bactericidal and virucidal properties that can be used as coatings in hospitals, public transportation and other institutions, to minimized the spread of infectious diseases. In this context, it is important to have a suitable photocatalyst with antimicrobial activity in the presence of visible light irradiation. TiO_2semiconductor and copper can be coupled to obtain a material with these desired properties [1][2]. In this study, we developed a new photocatalytic material through the heterojunction of commercial TiO_2nanoparticles with copper deposited by magnetron sputtering technique in distinct deposition times. Samples were characterized by Scanning Electron Microscopy, X-ray Diffraction, Raman Spectroscopy, Uv-vis Spectroscopy and Inductively Coupled Plasma/Optical Emission Spectrometry. To evaluate photocatalytic activity, degradation of Rhodamine B dye was quantified in presence of the samples, under irradiaton at 405 nm. Bactericidal activity was assayed with *Escherichia coli*at 405 nm and in dark conditions to obtain minimum inhibitory concentration (MIC) of tested samples. The characterization confirmed presence of anatase as major phase and copper in low concentration (< 5,0 % w/v). The degradation of Rhodamine B dye was effective under visible light conditions, as well as the bactericidal activity, with a MIC of 12,5 $mg.L^{-1}$for the sample with 30 minutes of copper deposition. The plasmonic nanostructures obtained will be further evaluated for virucidal activity and other light conditions.

Acknowledgements:
RHAE/CNPq, Plasmar Tecnologia and Epipolé-group

References:

[1] S. M. Imani and L. Ladouceur et al., ACS Nano, 14, 12341-12369 (2020)
[2] Y. Xin and K. Yu et al., Advanced Materials, 33, 2008145 (2021)

CuO Catalysts Prepared by Chemical Precipitation Synthesis for CO_2 Photoreduction Application

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The conversion of CO2 into fuels or chemical raw materials can be part of the solution to environmental and energy crises. The photocatalytic reduction of CO2 pollutant to substances of higher aggregated value can already be observed in a variety of studies. However, for this process to become viable, the development of catalysts that present high selectivity, long-term stability, and low overpotential is crucial. [1] In this context, CuO is a promising material that can convert CO2 into both alcohol and hydrocarbons. In this work, we studied the influence of adding different bases during the synthesis of CuO catalyst by the chemical precipitation route [2]. The studied bases were NaOH, KOH, monoethanolamine (MEA), and NH4OH. We selected MEA as a base with the aim of creating a surface functionalization on CuO catalyst, which can act as CO2 adducts andimprove the catalyst efficiency during photo or electroreduction processes. The synthesizedcatalysts were characterized via X-ray Diffractometry (XRD) and X-ray Photoelectron Spectroscopy (XPS), proving the CuO phase as the crystalline structure of all the samplesand the presence of nitrogen in the CuO/MEA catalyst surface. The use of different bases onthe synthesis of CuO by precipitation route has shown great influence in its photoactivity forCO2 reduction. The CuO/KOH and CuO/MEA samples presented enhanced gaseous production in comparison with samples CuO/NH4OH and CuO/NaOH. The CuO/MEA sample achieved the best photoactivity and selectivity, which was attributed to the surface functionalization by nitrogen groups.

Acknowledgments

CAPES (code 001), CNPq (processes 468956/2014-0 and 409904/2016-3), UFSCar, IFSP, and Embrapa Instrumentation. We also thanks Gelson T.S.Tavares, Cauê Ribeiro, Letícia V. Savazi, and Douglas M.S. Del Duque.

References

[1] Dong, W.J. et al. Nano Energy 78(2020)105168

[2] Nogueira, A. E. et al. Colloids and Surfaces A: Physicochem. Eng. Aspects 498(2016)161

Effect of fuel-to-oxidizer ratio on the synthesis of photocatalytic NiO by solution combustion method in a sucrose-nitrate system

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The growing world population and industrial development have caused concerns regarding energy crises and environmental damage. Solar energy and photocatalytic technology using semiconductor materials such as ZnO, TiO2, and NiO are promising solutions for the degradation and inactivation of chemical pollutants, and are clean, safe, and effective [1]. Nickel oxide (NiO), one of the most widely used transition metal oxides, has a wide band gap (3.0 - 4.0 eV) and can be synthed using cost-effective methods, such as solution combustion synthesis (SCS) [2]. SCS is a versatile method in which metal salts, water, and organic fuel are mixed and ignited by heat, resulting in oxide or metal nanoparticles (NPs). It is well known that the characteristics of the final product are strongly dependent on the amount of heat generated and gases released during the combustion reaction [3]. In this study, a nanocrystalline NiO photocatalyst was synthed by the SCS method using nickel nitrate as the oxidizer and sucrose as the fuel, by varying the fuel-to-oxidizer ratio (Φ) from fuel-leanto fuel-rich in a muffle furnace preheated at 500 °C. The effect of Φ on the SCS mechanism, morphology, crystallinity, surface area, and optical properties was investigated by SEM, XRD, BET/BJH, and UV-Vis spectroscopy. The photocatalytic activity was evaluated undervisible light using TC-HCL as the pollutant model. NPs synthed under fuel-rich conditions exhibited metallic Ni in addition to NiO. Under fuel-lean conditions, only NiO was observed,and the particles presented a higher surface area, resulting in better photocatalytic activity.

Acknowledgments:
This work was supported by CNPq, CAPES, FAPESC and PRH-ANP/FINEP

References:
[1] I. Ahmad et al., Adv. Colloid Interface Sci., vol. 311, no. December 2022, p. 102830 (2023)
[2] A. O. Juma et al., J. Alloys Compd., vol. 723, pp. 866-872 (2017)
[3] D. N. V. V. K. Lutukurthi, S. Dutta, and D. K. Behara, Ceram. Int., vol. 46, pp. 22419-22428 (2020)

Effect of relative band position between semiconductors in photoanodes dedicated to greenhydrogen production: TiO_2/CdTe case

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Green hydrogen is the focus of current research on renewable and sustainable fuels. For water splitting, TiO_2photoanodes (Eg^{bulk}= 3.2 eV) dedicated for oxygen generation are limited by the narrow solar radiation absorption window. Thereby, sensitization with CdTe quantum dots is of great interest due to the easy adjustment of the optical band gap and consequently the relative positioning of bands, largely controlled by the nanoparticle . Thus, the current work is dedicated to the study of energy transfer mechanisms at the interfaces of TiO_2/CdTe semiconductors as a function of the relative positioning of bands of the two semiconductors. TiO_2nanotubes supported on Ti foils were produced by sonoelectrochemical strategy.[1] The CdTe quantum dots stabilized by 3-mercaptopropionic acid (MPA) were produced via electrochemistry, from the in situ reduction of Te^0.[2] The control of CdTe was done through different heating times leading to tuning from green (λ_{em}= 529 nm, Eg = 2.40 eV) to red (λ_{em}= 643 nm, Eg = 1.98 eV). The system has a higher luminescence quantum yield (QY) for yellow nanoparticles (24.96%). The best sensitization times of TiO_2NTs and CdTe-MPA were evaluated using electrochemical techniques such as chronoamperometry, linear voltammetry and electrochemical impedance spectroscopy. The results indicated that6 hours is the best sensitization time of TiO_2, and the heterojunction made by TiO_2/CdTe. The best electrocatalysis performance was for yellow CdTe, presenting photocurrent densityof 1.80 mA/cm^2, showing an increase of 9x in relation with pure TiO_2. The obtained results demonstrate the material potential to application in hydrogen production.

References

[1] J. Power Sources 506 (2021) 230165
[2] Dyes and Pigments, 180, 2020, 108483

Acknowledgments
MCTI, CNPq, FINEP, FUNDEP.

Light-driving photoconversion of CO2 into Solar Fuels

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In recent years, the increase of carbon dioxide (CO2) in the atmosphere has become a global environmental issue because of the serious problems, such as the “greenhouse effect”. The idea of mimicking the overall natural photosynthetic cycle of chemical conversion of CO2 into useful fuels has been consistently gaining attention for more than thirty years. Such artificial photosynthesis allows direct conversion of CO2 and water on photocatalysts into valuable hydrocarbon using sunlight at room temperature and ambient pressure to serve to reduce atmospheric CO2 concentrations while providing on a renewable carbon fixation and energy storage. In this presentation, we will report the utilization of solar energy to highly efficient conversion of CO2 into renewable hydrocarbon fuel over structured nanomaterials. The geometric shape and exposure of specific crystal planes of the nanostructures proves a requisite for the high level of photocatalytic reduction of CO2 and formation of C2+ species.

References

[1] Y. Shen, Z. G. Zou, Y. Zhou, Nat. Commun. 14, 1117 (2023). [2] W. Gao, S. Li, Y. Zhou, Z. G. Zou, Nat. Commun. 12, 4747 (2021). [3] Q. T. Han, Y. Zhou, Z. G. Zou, Adv. Mater. 33, 2006780 (2021). [4] Q. T. Han, Y. Zhou, Z. Zou, J. Am. Chem. Soc. 141, 4209 (2019).

Synthesis and Characterization of Various TiO_2NTs/rGO Heterostructures and its Application for Photocatalytic Degradation of the Methylene Blue.

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The advantages of Titanium Dioxide (TiO_2) in nanobiotechnology are its non-toxicity, availability, cost-effectiveness, wide bandgap semiconductor, good biocompatibility, stability, and physical and chemical properties. TiO_2nanotube arrays are particularly useful for photocatalysis processes for pollutant degradation, H_2generation, and self-cleaning properties. However, TiO_2has a restricted photocatalytic efficiency due to its wide bandgap (3.2 eV), which limits the amount of solar radiation it can efficiently absorb in 4-7%. To improve the efficiency of TiO_2, researchers have explored several methods, including the addition of graphene oxide (GO) as a co-adsorbent. The GO has a high specific surface area and strong light absorption property that can improve the photocatalytic efficiency of TiO_2, but it also contains a large number of oxygen-containing functional groups that reduce its electronic properties. Therefore, researchers need to modify the GO to reduced graphene oxide (rGO) to recover its charge carrier mobility and reduce the number of oxygen-containing functional groups. Hence by these facts, this research investigates the performance of TiO_2/rGO NTs in photocatalytic degradation of the methylene blue, as well as the properties of the produced GO and the various heterostructures of TiO_2/rGO NTs, produced by a low-cost thermal reduction at a different range of temperatures (400/420/450/470/500/520°C) for 2 and 5 hours, using various analysis techniques, such asUV-Vis spectroscopy, XDR, Raman, FTIR, SEM, TGA, DTG, DSC, and zeta potential analysis.

Magnetic activated carbon for improving the removal of antibiotics from water by heterogeneous solar photo-Fenton

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Activated carbon (AC) has been considered as a promising support for Fe-based solid catalysts in heterogeneous Fenton oxidation, due to its low cost and environmentally friendly properties [1]. However, few studies have investigated the application of AC-derived catalysts in photo-Fenton for pharmaceutical removal. Therefore, this study aimed to develop a magnetic activated carbon-based catalyst (MAC-Fe) and evaluate its efficacy in removing antibiotics sulfamethoxazole (SMX) and trimethoprim (TMP) from water. The MAC-Fe catalyst was produced by impregnating KOH and $FeCl_3$in one step, a simplified and potentially cost-reducing process that combined the chemical activation and magnetization into a single step. The determination of pore morphology and specific surface area (SBET) showed that MAC-Fe has a suitable microporous structure (~16% micropore volume) and reasonable SBET (41 $m^2 g^{-1}$). Also, high saturation magnetization (Ms) of 21.14 emu g^{-1}exceeds most reported magnetic-activated carbons [2]. To evaluate the real contribution of MAC-Fe in the removal of antibiotics, the ability to remove SMX and TMP were studied with 250 mL of a mixed solution containing 200 μg $L^{-1}$of SMX and TMP, MAC-Fe 0.25 g L^{-1}, $H_2O_2$5 mM at pH 8.0. The results of MAC-Fe application for antibiotic removal suggested that the photo-Fenton and Fenton-heterogeneous processes were the most effective in eliminating the antibiotics, followed by adsorption. In the whole removal process, adsorption can serve as the initial step, whereby the MAC-Fe adsorbs antibiotics and H_2O_2onto its surface. Overall, this study suggests that using MAC/Fe in heterogeneous photo-Fenton processes is an effective strategy for removing SMX and TMP from water.

Acknowledgments:
CAPES - Finance Code 001.

References:

[1] Shang J. G, Kong X. R, He L. L, Li W. H, Liao Q. J. H. Int J Environ Sci Technol., 13, 2449-58 (2016).

[2] Rocha L. S, Pereira D, Sousa É, Otero M, Esteves V. I, Calisto V. Sci Total Environ., 718. (2020).

Microwave-assisted solution combustion synthesis of N-doped zinc oxide nanoparticles with enhanced photocatalytic activity for water remediation

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Semiconductor nanoparticles can be made available by solution combustion synthesis (SCS), a cheap, simple, scalable and very fast method that has been used for a wide range of applications [1]. This method, conventionally carried out in muffle furnaces, has been modified so that the reaction is initiated by microwave irradiation, called Microwave Assisted Solution Combustion Synthesis (MASCS) [2]. Such a modification resulted in much faster heating and cooling rates compared to conventional SCS, thereby increasing the number of defects on the nanoparticle surface, narrowing the band gap, reducing the particle size and increasing the porosity of the synthesis product. Unlike previous reports in the literature [1-3], this work used a mixture of fuels (sucrose and urea in a molar ratio of 1:1) in solution to achieve a one-step synthesis and doping of the ZnO nanoparticles. The doping effect - confirmed by XPS, XRD and UV-Vis spectroscopy - greatly improved the ability of the developed material to remove tetracycline from water under visible light photocatalytic assays, where the doped materials showed over 90% pollutant removal in 120 minutes.

Acknowledgements:

The authors would like to thank CNPq, CAPES, and FAPESC for the financial support to this work.

References:

[1] Z. Kalantari Bolaghi, S.M. Masoudpanah, M. Hasheminiasari, Photocatalytic properties of ZnO powders synthesized by conventional and microwave-assisted solution combustion method, J. Sol-Gel Sci. Technol. 86 (2018) 711-718.

[2] L.C. Nehru, C. Sanjeeviraja, Microwave-Assisted Combustion Synthesis of Nanocrystalline ZnO Powders Using Zinc Nitrate and Various Amount of Organic Fuels as Reactants: Influence of Reactant Parameters - A Status Review, Nano Hybrids. 6 (2014) 75-110.

[3] N. Assi et al., Synthesis and characterization of ZnO nanoparticle synthesized by a microwave-assisted combustion method and catalytic activity for the removal of ortho-nitrophenol, Desalin. Water Treat. 54 (2015) 1939-1948.

NaNbO3/Eumelanin composite: A new photocatalyst under visible light

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Research using semiconductors as photocatalysts has been carried out since the 70s. In this photocatalytic process, photons with energy equal to or greater than the band gap energy absorbed by the semiconductor, generate electron/hole pairs, thus creating oxidizing and reducing sites in its surface. Many factors affect the activity of photocatalysts such as particle size and optical absorption capacity. Sodium niobate (NaNbO3) is a promising photocatalyst however, the natural shape of particles are large cubes, but research has already reported nanoparti-cles with high photocatalytic activity [1]. In addition, it only absorbs ultraviolet light, limiting its use. Therefore, it is highly desirable to extend the optical absorp-tion into the visible range. Eumelanin is a pigment present in abundance in fau-na and flora, responsible for the black-brown color. It has many physicochemical properties, of particular interest here broad optical absorption ranging from UV to near infrared and strong affinity by metals [2]. Given the above, we present a rapid method to synthesize NaNbO3 nanoparticles. These nanoparticles were used to prepare for the first time a NaNbO3/Eumelanin composite with photocatalytic ac-tivity under visible light. The photocatalytic activity was analyzed through the deg-radation of Rhodamine B (RhB), a dye widely used in industry and highly toxic. The results showed that pure NaNbO3 and pure Eumelanin do not show photo-catalytic activity, while the composite was able to successfully degrade the dye. Therefore, this study presents an alternative photocatalyst that uses visible light and widely available elements, serving as inspiration for the development of new photocatalysts.Acknowledgements: CAPES, CNPq, FAPERGS, UNIFESP Campus São José dos Campos e UNIPAMPA Campus Bagé.References:[1] D. Fernandes et. al., Ceramics International vol. 47, 10185 (2021).[2] W. Xie et. al., ACS Sustainable Chemistry Engineering vol. 8,1343 (2020).

Optimization of niobium-based materials for photocatalysis and solar energy conversion

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This work presents a study on the optimization of materials for use in photocatalysis and solar energy conversion. Niobium-based materials such as Nb2O5, NaNbO3, and KNbO3 have been shown to improve the efficiency of these devices. In this study, we have used Density Functional Theory (DFT) calculations to determine the most suitable dopants to reduce the bandgap and enhance the performance of these materials. The optimized materials have been obtained through theoretical and experimental studies. Our results demonstrate the significant improvement in the efficiency of these devices with the optimized niobium-based materials. This research contributes to the development of more efficient materials for use in renewable energy applications, such as solar cells and photocatalysts.

Optimizing sucrose-urea fuel mixture in solution combustion synthesis of N-doped zinc oxide nanoparticles for enhanced photocatalytic removal of tetracycline hydrochloride

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Recent data have revealed the detection of approximately 160 emerging contaminants in Brazil and 60 in different regions of the world, which can cause adverse or lethal problems [1]. Semiconductor-based photocatalysis is a green technology for the environmental remediation of these substances. Zinc oxide (ZnO) has been applied as a photocatalyst with proven activity for pollutant abatement [2]. This research aims to design and manufacture nitrogen-doped zinc oxide nanoparticles using a one-step approach by tailoring the molar ratio of urea-to-sucrose as fuels in a mixture with zinc nitrate hexahydrate as the zinc precursor. Five samples were prepared to vary the molar ratio in fractions of 0%, 25%, 50%, 75%, and 100% of urea, in a heated Muffle furnace at 500 ºC for 10 min. Physicochemical properties of the N-doped ZnO were investigated through XRD, SEM, DLS, BET/BJH, XPS, and UV/vis. Photocatalytic removal of the model emerging pollutant tetracycline hydrochloride (TC - HCl) was performed under light (15 W, λ >420 nm) with the N-ZnO as photocatalysts. The results reveal the feasibility of producing one gram of ZnO in a short time. Nano-ZnO with a urea-to-sucrose molar ratio of 50% had a surface area of 26 m^2/g, mesoporosity, and a band gap of 2.89 eV. These features enhance the photocatalytic removal of the TC - HCl to around 75% for 60 min of irradiation, which means an efficiency improvement of 40% and a removal rate 1.86 faster than the ZnO undoped. After 120 min, the photocatalytic degradation of TC - HCl is almost 100% due to the synergy of material's porous structure and a narrow band gap.Keywords: N-doped zinc oxide; solution combustion synthesi photocatalysi visible light active.

Acknowledgments: The authors would like to thank CAPES and CNPq for funding this study.

References

[1] M. .C.V.M. Starling et al., Journal of Hazardous Materials, 372, 17 - 36, 2019.

[2] O. Baaloudj, Journal of Water Process Engineering, 42, 102089, 2021.

Photoanodes Based on Heterojunction of $CuBi_2O_4/TiO_2$for Green Hydrogen Production

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Green hydrogen emerges as a great candidate for obtaining clean and renewable energy and its photoelectrochemical production is one of the frontiers of research. Titanium dioxide (TiO_2) stands out compared to other semiconductors due to its stability[1]. However, its bandgap (Eg = 3.2 eV) does not allow absorption of the entire solar spectrum. Thus, heterojunctions that allow increased radiation absorption and efficient transfer are of research interest. Copper spinels and bismuth $CuBi_2O_4$(CBO) can act as a TiO_2sensitizer for photoelectrochemical conversion of solar radiation to green hydrogen, given its significant spectral absorption region and positive flat band[2]. The present work was dedicated to the electrodeposition of BOD on the nanostructured surface of $TiO_2$and the characterization of the photoelectrochemical performance of photoanodes prepared for OER. From supported TiO_2nanotubes, $Cu^{2+}$and Bi^{3+}ions were deposited, evaluating the effect of electrical chargeson chronopotentiometric processes and ion concentration (in dilution factors of 50-100x).After electrodeposition, the BOD samples were dried and thermally treated at 500◦C. The materials produced were structurally characterized by X-ray diffraction, verifying the formation of the crystalline phases of interest for spinel BOD and TiO2. Scanned electronmicroscopy images showed the evolution of surface micro- and nanostructures. The photoelectrochemical characterizations allowed access to the electrochemical performance data of the produced photoanodes. Based on the experimental design (load x concentration), the best condition can be determined leading to a photocurrent density of around $1mA/cm^2$, under simulated solar irradiation (filter AM1.5G, 100 mW. cm^{-2}, S^{2-}/SO_3^{2-}, pH 13) a 4x performance increase over pure TiO_2.

Thanks to:
MCTI, FACEPE, PGMTR-UFPE.

References:
1Velasquéz, J. Power Sources 506 (2021) 230165
2Oropeza, et al. Journal of Physical Chemistry C 2020 124 (41), 22416-22425.

Pulsed laser ablation of gold in water for the fixation of CO2 into advanced functional nanomaterial

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The CO_2reduction reaction is up today exploited for the synthesis of value-added feed-stocks in gaseous or liquid form [1]. Here we report an example of CO_2reduction reaction leading to the production of advanced functional nanomaterial by the use of pulsed laser ablation ofa gold target in water. The presence of CO_2derivatives in water during the ablation process, leads to C2 and C3 coupling with the production of organic material with the typical fingerprint of the CO_2reduction reaction. Carbon monoxide rich gold nanoparticles and metal-organic nano-frameworks composed by gold nanoclusters and carboxylic acid crystals, are produced by the catalytic reactions induced during pulsed laser ablation, together with metal-free carboxylic acids and residual carbon nanomaterial in tubular and onion like structure. Using a novel pump and probe configuration for the pulsed laser driven CO_2reduction reaction, we are able to obtain ultra-small AuNPs (d < 3 nm) and photoluminescent metal-organic nano-framework with a quantum-yield of the about 23% in the blue spectral region [2]. The authors acknowledge CAPES, CNPq and FAPERJ for the support.

[1] F. Creutzig et Al., "The mutual dependence of negative emission technologies and energy systems", Energy Environ. Sci., 2019,12, 1805-1817
[2] A.R Ziefuss et Al., "Photoluminescence of Fully Inorganic Colloidal Gold Nanocluster and Their Manipulation Using Surface Charge Effects", Adv. Mater. 2021, 2101549

Removing antibiotics from water via solar photo-Fenton using cobalt containing magnetite

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Fenton-type reactions are effective for the generation of hydroxyl radicals which can degrade contaminants in wastewater. Magnetite contains Fe(II) and Fe(III) species that areimportant in the catalytic activity of the Fenton reaction. Furthermore, the iron in the magnetite structure can be replaced by other metals, such as cobalt, improving the catalyticproperties of the material [1]. In this work, the influence of cobalt on magnetite structuretowards the Fenton process was studied, where $Fe^{2+}$was replaced by Co^{2+}in previously chosen stoichiometric. The photo-Fenton experiments were conducted under solar irradiation, from 11:00 am to 1:00 pm, in an open glass reactor with a maximum capacity of 300 mL. The catalytic activity of magnetites was evaluated based on the degradation of sulfamethoxazole and trimethoprim. Although cobalt ions incorporation on magnetite structure showed no significant difference on degradation after 90 min in the dark, complete degradation of both antibiotics was achieved after 45 min under solar irradiation (86 kJ m^{-2}accumulated UVA dose) using $[Co_{0.25}Fe_{0.75}][Fe_2]O_4$, the most efficient catalyst. Unexpectedly, $[Co_{0.25}Fe_{0.75}][Fe_2]O_4$was the magnetite with the lowest specific area (144 m^2g^{-1}). Cobalt exhibit the redox pair Co^{2+}/Co^{3+}, which can produce radicals according to the reaction $Co^{2+}+H_2O_2\rightarrow Co^{3+}+OH^-+{}^{\cdot}OH$. Also, the reduction of $Co^{3+}$by $Fe^{2+}$is thermodynamically favorable ($Fe^{2+}+Co^{3+}\rightarrow Fe^{3+}+Co^{2+}$, $\Delta E=1.04$ V), by an electron transfer process within the oxide structure [1]. This finding indicate that the higher catalytic activity of $[Co_{0.25}Fe_{0.75}][Fe_2]O_4$can be attributed to the cobalt incorporation, which plays a more important role in the photo-Fenton processes than the specific area of the catalyst under the conditions applied.

Acknowledgements: FAPESP, INCT-CNPq, CAPES and PROPe-PROPG/UNESP.

References:[1]Costa,R.C.C.;Lelis,M.F.F.;Oliveira,L.C.A.;Fabris,J.D.;Ardisson,J.D.; Rios, R.R.V.A.;Silva, C.N.;Lago, R.M. 129, 171-178 (2006).

Self-cleaning membrane made of cellulose and MIL-88A for the treatment of contaminant emulsions

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The water shortage crisis has pressured industries to adopt water reuse practices. Self-cleaning membranes are a promising solution for effluent treatment, followed by the regeneration of the used membrane clogged with contaminants. In this work, a prepared cellulose-MIL88A membrane presented 12 cycles of filtration and regeneration, removing 96.3 % of RB5 dye and 90 % of total organic carbon. The regeneration of the membrane was performed via degradation of the contaminant using the photo-Fenton process and MIL88A as the photocatalyst. The membrane was able to separate emulsified contaminants in water such as diesel fuel. These results suggest that the proposed membrane is promising for water reuse from effluent treatment.

[1] Biswas, A. K., Tortajada, C. (2019). Water crisis and water wars: myths and realities. International Journal of Water Resources Development, 35(5), 727-731.

[2] Haarstrick, A., Bahadir, M. (2022). Water and its Global Meaning. In Water and Wastewater Management: Global Problems and Measures (pp.3-14). Cham: Springer International Publishing.

[3] Khandelwal, G., Raj, N. P. M. J., Vivekananthan, V., Kim, S. J. (2021). Biodegradable metal-organic framework MIL-88A for triboelectric nanogenerator. Iscience, 24(2), 102064.

[4] Lin, K. Y. A., Chang, H. A., Hsu, C. J. (2015). Iron-based metal organic framework, MIL-88A, as a heterogeneous persulfate catalyst for decolorization of Rhodamine B in water.RSC Advances, 5(41), 32520-32530.

[5] Gao, J., Ma, S., Xu, M., Yuan, M., Li, J., Xue, J., Wang, M. (2022). Photo-Fenton superwettable NiFe2O4/TA/PVDF composite membrane for organic pollutant degradation with successively oil-in-water separation. Chemosphere, 286, 131705.

STRUCTURAL, CHEMISTRY, AND ELECTRONIC STATEOF THE INTERFACES OF TRANSPARENT CONDUCTING OXIDE AND HEMATITE APPLIED IN PHOTOELECTROCATALYSIS

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Photoelectrocatalysis devices play a key point in green power generation systems. At the nanoscale, the efficiency of the device is driven by the interface [1,2], which can be assessed by HRTEM images, in addition to EELS and EDS coupled to a scanning mode microscope (STEM) [3]. In this study, we investigated the interface of hematite with 3% Zr deposited on fluorine-doped tin oxide, and we added a NiFeOx solution to form a layer of co-catalytic material (Ni). Samples were prepared using a FIB. We obtained chemical and electronic state characterization at the interface via STEM-EELS-EDS maps. The results show a 2.5-nm thick interface containing a Fe-Sn-O mixture. Structural analysis of the interface by HRTEM imaging showed that the interface exhibited lattice distortions and high stress. The concentration map revealed that Zr segregated at the grain boundaries of the hematite and in the mixing region of the interface and that Ni is deposited on the free surface of the hematite. The energy maps show a shift towards Sn+4 to Sn+2 and Fe+3 to Fe+2 for both FTO and Hematite grain boundaries, caused by the loss of oxygen from the system.

Acknowledgments
The authors acknowledge CNPq, CAPES and FAPESP. G.T.S acknowledge CAPES (Gant 88882.438914/2019-01). K.C.B., T.E.R.F and I.R acknowledge FAPESP (Grant 19/06654-9, 21/07459-5, and 21/10672-2). J.B acknowledge CNPq (315350/2020-2).

Synthesis and characterization of iron (III) oxide/bismuth molybdate thin film heterostructure forthe photo electrocatalytic degradation of antibiotic oxytetracycline.

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In this work, nano-heterostructures based on iron (III) oxide/bismuth molybdate (α-Fe2O3/Bi2MoO6) thin films were fabricated by a dip-coating technique using aqueous solutions. The heterostructures were deposited on fluorine-doped tin oxide glass substrates. From a detailed characterization using Fourier transform-infrared and X-ray diffraction, the formation of α-Fe2O3 (hematite) and bismuth molybdate was demonstrated.

The field emission scanning electron microscopy cross-section images confirm the formation of a well-defined α-Fe2O3 layer under the Bi2MoO6 layers. The optical band gap energies for the heterostructures obtained were estimated from the diffuse reflectance spectra and correspond to 1.6 eV.

The photoelectrochemical degradation of the antibiotic oxytetracycline (OTC) at different pH values was investigated in an aqueous solution under visible and solar light irradiation. Particular emphasis was given to the kinetics and mechanism during the photocatalytic degradation of OTC.

Moreover, the mechanism of photochemical degradation of OTC was investigated at different potential values under solar light.

Synthesis of MoS2/MoO3 composites for the removal of cationic dyes by adsorption process

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Dyes are used in a diversity of industries, such as plastic, textile, paper, and others. The annual production of synthetic dyes worldwide is about 7.107 tons and around 10,000 tons are being used by textile industries. Textile dyes are persistent pollutants that can bring several threats to human health and the environment.[1,2] In this context, the application of an adsorbent to remove dyes from contaminated wastewater is an interesting method that does not involve drastic changes in the usual water treatment plant. In the present work, we studied the synthesis and application of MoS2/MoO3 composite for the adsorption of the methyl blue(MB) organic dye. The synthesis of the samples was performed by the calcination of the MoS2 precursor under different temperatures and calcination times to evaluate their adsorption efficiency and find the best synthesis conditions. Samples were characterized via thermogravimetry (TG), X-ray Diffractometry (XRD), infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). As result, it was possible to notice efficient adsorption properties in the materials calcinated under 300 ºC. XRD measurements confirmed the MoS2/MoO3 composite formation, while FTIR and TG analyses showed that it occurs only on the surface of the adsorbent particles. SEM images with EDS presented a uniform distribution of Mo, O, and S on the sample's surface. Finally, Isotherm adsorption measurements conducted with the main sample attained the maximum adsorption of 505.1 mg of MB adsorbed per g of adsorbent, which is a great value compared with the literature. In summary, the studied materials presented attractive adsorption properties under mild temperature and time synthesis conditions.

Acknowledgments

CAPES(code 001), CNPq(processes 468956/2014-0 and 409904/2016-3), UFSCar, IFSP, and Embrapa Instrumentation.

References

[1]Al-Tohamy, R. et al. Ecotoxicology and Environmental Safety 231 (2022) 113160

[2]Chandanshive, V. et al. Chemosphere 252 (2020) 126513

Titanium dioxide photoanodes sensitized with bismuth sulfide quantum dots for hydrogen production

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Brazil has one of the cleanest energy matrices in the world. The solar irradiation predominant in the Brazilian Northeast makes the region one of the most promising in the exploitation of solar energy for conversion into electrical energy. Additionally, thanks to the intermittent character of the sun, it is necessary to store energy in the form of a vector, making it possible to use it in the absence of its main source.[1] Hydrogen, therefore, is one of the main fuels and enables carbon-free combustion, which benefits the environment. In this work, titanium dioxide (TiO2), a semiconductor with a band energy of 3.2 eV, is applied as a photocatalyst in the production of photoanodes using the spin-coating technique.[2] Films are produced over FTO in two steps. A TiO2 compact layer is deposited by adding titanium diisopropoxide and thermal treatment at 500°C for 60 min. The TiO2 mesoporous layer was optimized in terms of paste concentration and the number of deposited layers, and at the end of the process the photoanode was thermally treated at 450°C for 30 min. The photoelectrochemical performance of the photoanodes was linearly dependent on the layer thickness, with a maximum value of 0.34 mA.cm-2 with a thickness of 4.20 ± 0.17 μm afterthe deposition of 4 layers in a ratio of 1:4 (TiO2:EtOH). In order to broaden the spectrumregion, bismuth sulfide nanocrystals were applied as sensitizers, with a band energy of 1.9eV. After sensitization, performed through chemical adsorption for 24 h, the photoanodes showed 1.5 mA.cm-2, a value 4.4 times higher than pure TiO2. Thus, photoanodes have great potential for photoelectrochemical green hydrogen production, proving the concept of improving photoelectrochemical performance by sensitizing mesoporous TiO2 films with semiconductor nanocrystals.

[1]J.H. kim et al.,Chem. Soc. Rev.,2019,48,1908-1971.
[2]D. Velasquez et al.,J. Power Sources,2021,506,230165.

Use of immobilized microalgae for effluent treatmentand concomitant biomass production

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The use of microalgae for effluent treatment has gained prominence in recent decades. Therefore, new technologies and processes have shown satisfactory results regarding the efficiency of bioremediation. One of the options used is based on the immobilization of microalgae in polymeric matrices, which aims to optimize the intrinsic characteristics of these microorganisms for the treatment of wastewater [1]. Thus, the objective of this work is the development and characterization of a polymeric matrix based on alginate and nano-chitosan for microalgae cultivation and effluent treatment, as well as evaluating its bioremediation potential. The methodology consists of trapping the cells inside a porous and semipermeable membrane in the form of a capsule, through physicochemical interactions. The cell immobilization system is an excellent alternative to traditional treatment methods, as it aims to increase efficiency and reduce costs in processes, decreasing energy consumption in unit operation, enabling cell reuse and significantly increasing the capacity for complexation and pollutant treatment [2]. Furthermore, it is possible to simultaneously treat wastewater and produce microalgae biomass, reducing the concentration of pollutants by approximately 80% and achieving yields of up to 1.6 g/L of biomass. From the biomass, various high-value-added bioproducts can be obtained, especially those related to biofuel production. This process is relatively new and still has fields to be explored and possibilities for technological implementations.

Acknowledgments:

To UFPR, CAPES, and the advisors professors.

References:

[1] KUBE, M.; FAN, L.; RODDICK, F. Algal Research, v. 54, (2021).

[2] DA SILVA, P. A. S. et al. Waste to Energy, v. 1, p. 511-537, (2022).

Visible Light Photoelectrochemical Sensor for Dopamine: Determination Using Iron VanadateModified Electrode

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This study presents a simple, cost-effective method for producing iron vanadate semiconductor material with remarkable electroactivity.[1,2] This material was used as a photoelectrochemical sensor for detecting dopamine. The iron vanadate obtained using the Successive Ionic Adsorption and Reaction process was thoroughly characterized in terms of its structure and morphology. Photoelectrochemical characterization showed that the photoanode had high photoelectroactivity when irradiated with visible light. Under optimal conditions, dopamine was detected using chronoamperometry at +0.35 V (vs. Ag/AgCl), achieving two linear response ranges (1.2-30.3 and 30.3-72.8 μmol L^{-1}). The limits of detection and quantification were 0.34 and 1.12 μmol L^{-1}, respectively. The accuracy of the proposed electrode was evaluated by measuring dopamine in artificial cerebrospinal fluid, resulting in recovery values ranging from 98.7 to 102.4%. The selectivity of the sensor was also tested by detecting dopamine against several interferent species, and the results demonstrated good precision and promising potential for the proposed method. Additionally, electronic structure calculations based on DFT were performed to aid in interpreting the results. The dominant dopamine species were identified under the experimental conditions, and their interaction with the iron vanadate photoanode was proposed. The improved light-induced dopamine detection was also evaluated regarding the charge transfer process.

Acknowledgment:

CAPES, LMEM, INCT-Bio, and ESPEC.

References:

[1] M. R. S. Pelissari, L. P. Camargo, et al., J. Mater. Sci., 57, 7173-7190 (2022).

[2] M. M. Sajid, H. Zhai, et al., Mater. Sci. Semicond. Process., 129, 105785 (2021).

Application of tungstate in photocatalysis of pollutant

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Tungstates have aroused great interest in recent decades, as they play a key role in emerging technologies for applications in sectors ranging from the environmental area through the development of new catalysts, to the energy area with the development of materials with optical properties for application in lighting devices in the solid state. Recently, the increase in the level of pollution around the world has not only contributed to environmental issues, but also aroused considerable interest in photocatalysis that act as promising treatments, based on studies concerning tungstate. The objective of the study in question refers to the applicability of tungstate of various types with the property of environmental treatment by means of pollutant controls, demonstrating its efficiency with respect to the improvement of the environment. The information obtained was based on searches made through databases with the use of specific keywords in order to filter as much as possible the possible results directing them to the theme, observing the data, it is noted the increasing use of tungstate for various applications through time. It is concluded then that a study regarding the application of tungstate in photocatalysis of pollutants becomes very promising and innovative due to its vast area of applications with respect to the health of the planet as a whole.

Application of ZnO-SiO2 oxides in the degradation of Aldrin pesticide by heterogeneous photocatalysis

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The environment is continually impacted by water sources, and one of the main pollutants is agricultural and industrial waste. In this context, pesticides are widely used in the agricultural sector and due to their toxic nature, chemical characteristics and because they are biologically stable, their degradation has been one of the great challenges for the scientific community [1]. Thus, the present work aims to investigate the catalytic activity of the binary oxides of ZnO-SiO2 obtained by the Pechini method, aiming at application in the photocatalysis process for the degradation of the Aldrin pesticide. The materials obtained were characterized by X-Ray Diffraction (XRD), Infrared (IR), UV-Visible and Raman Spectroscopy, BET and Scanning Electron Microscopy (SEM). The photocatalytic tests were carried out in a photocatalytic reactor with UVC lamps with a reaction time of 1 to 4 h. The oxides were successfully obtained, according to the DRX standards and the ICDD charts of ZnO and SiO2. In relation to the IR analysis, it was possible to observe characteristic molecular vibrations of SiO2(490 cm-1) and ZnO(430 cm-1), and from the absorption spectra in the UV-visible region it was possible to calculate the band gap of the catalysts the values obtained varied between 2.88-2.92 eV. In the Raman spectra, we observed a greater degree of symmetry for samples containing a greater amount of Si4+, however, the FWHM values calculated from XRD revealed a lower degree of long-range symmetry for samples with a smaller amount of Si4+. As for the photocatalytic tests with the pesticide Aldrin, the samples Zn0.90Si0.100, Zn0.80Si0.200 and Zn0.70Si0.300, presented 31.6; 42.3 and 56.4% degradation, respectively.

Acknowledgment: UEPB/PRPGP/PPGQ FAPESQ/PRONEX, and NPE-LACOM/UFPB

[1] PIGNATELLO, Joseph J.; OLIVEROS, Esther; MACKAY, Allison. 36, 84, (2006).

Babassu mesocarp-based TiO_2 for photocatalytic application

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A composite based on the green polysaccharide mesocarp of babassu and $TiO_2$was synthesized through the sol-gel technique [1,2]. XRD, UV-Vis Diffuse Reflectance Spectroscopy-DRS-Uv-Vis, and N_2adsorption-desorption characterized the synthesized material. Photocatalytic tests were carried out with the dye methylene blue (MB) (concentration 12.0×10^{-6}mol L^{-1}) under UV-Vis light, the samples were removed in time determinate and were centrifuged at a rotation of 5000 rpm. Subsequently, they were analyzed by a spectrophotometer UV-Vis in a 200 to 800 nm. The dye concentration was determined using the absorption band at 664 nm. XRD showed that the material presented the anatase crystallographic phase of TiO_2. The material presented a crystallite size of 8.01 nm, calculated by the Scherrer equation. From DRS-Uv-Vis, it was possible to calculate the band gap energy value of the material by the Kubelka-Munk method, obtaining the value of E_g= 3.19 eV. From the N_2adsorption-desorption technique, the BET method calculated the specific surface area, obtaining a value of 95.41 m2 g−1. The pore volume and average pore diameter were calculated by the BJH method, obtaining values of 0.11 $cm^3g^{-1}$and 4.6 nm, respectively. The photocatalytic test showed that the composite promoted an MB discoloration of 69.34%. It was concluded that the material synthesized from the green polysaccharide and $TiO_2$is promising in the proposed application.

Acknowledgments:

UFPI, LIMAV, CAPES, FAPEPI, CNPQ.

References:

[1] Araujo, F.P.; Trigueiro, P.; Honório, L.M.C.; Furtini, M.B.; Oliveira, D.M.; Almeida, L.C.; Garcia, R.R.P.; Viana, B.C.; Silva-Filho, E.C.; Osajima, J.A. Dalt. Trans., 49, 16394-16403 (2020).

[2] Lopes, A. C. B., Araújo, F. P., Morais, A. I. S., de Lima, I. S., Honório, L. M. C., Almeida, L. C., Garcia, R. P., Silva-Filho, E. C., Furtini, M. B., Osajima, J. A. Materials., 15(13) (2022).

$BiVO_4$ and V_2O_5 synthed by microwave-assisted hydrothermal method for selective adsorption towards methylene blue

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Dyes from industrial processes are potential sources of water pollution due to their aromatic structures and species loads, which can make their removal difficult. For this, adsorption processes with a solid material stand out, and the adsorptive efficiency will depend on the chemical nature, stability, charge, and surface of the material [1]. Thus, this work aims to synthe oxides in different proportions of bismuth and vanadate to apply them for the adsorption of methylene blue cationic dye (MB), in addition to testing the selectivity of oxides with methyl orange anionic dye (MO) and monitoring by UV/VIS spectrophotometry. For the synthesis of $BiVO_4$, two solutions were prepared, one with 8 mmol of NH_4VO_3in NaOH and the other with 8 mmol of $Bi(NO_3)_3.5H_2O$ in HNO_3. $V_2O_5$was synthed using 8 mmol of NH_4VO_3in NaOH and HNO_3[2]. The synthesis of the $BiVO_4/V_2O_5$composites occurred with a mol excess (1 and 5%) of NH_4VO_3. The mixtures were submitted to a hydrothermal reactor under microwave at 160°C/15 min. For the adsorption, AM 4 $\mu molL^{-1}$(pH 8) and 0.2 g $L^{-1}$of oxide synthed in the solution were used. The results showed that a higher proportion of bismuth in the Bi/V molar ratio did not significantly alter the adsorptive capacity of these materials. On the other hand, a higher proportion of vanadium in the Bi/V ratio and even the absence of bismuth (V_2O_5) significantly increased the adsorptive capacity. For the selectivity test, a solution with MO and MB was used. We conclude that the synthed $V_2O_5$was more efficient in the adsorption and desorption of the methylene blue dye and was selective when applied with MO, properties that were not observed for the samples rich in bismuth and commercial activated carbon.

Acknowledgements:

To LADEMA, GMPC, LMEM, LARX, Fundação Araucária, CAPES e CNPq.

References:

[1] Bagheri, A. et al. J. Environ. Chem. Eng., vol. 9, n. 1, p. 104879 (2021)

[2] Packiaraj, R. et al. J. Mater. Sci. Mater. Electron., vol. 29, n. 15, p. 13265 (2018)

Boosting the photocatalytic activity of ZnWO4 by ZnO nanoparticles incorporation

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The use of semiconductor materials and strategies to improve their photocatalytic properties have been widely explored. And, because of its chemical stability, optical, electronic properties, zinc tungstate (ZnWO4) has emerged as a promising photocatalytic material. Despite being a good semiconductor, with Eg = 3.8 eV, the electronic characteristics of ZnWO4 limit to the light absorption in the UV region, making it a restricted photocatalyst [1]. To improve the photocatalytic activity of ZnWO4, we aimed to decorate the ZnWO4 with different ZnO nanostructures. It has been reported that the particles morphology has an important role in photocatalysis [2]. The synthesis of these materials was done, for the first time, by a combination of methods that are: the Pechini-modified method and solvothermal synthesis. A proportion of 10 %wt of ZnO was fixed to be incorporated on ZnWO4. According to X-ray diffraction (XRD), infrared (IR) and ultraviolet-visible (UV-vis) spectroscopy and zero charge point analysis (PCZ), the target ZnWO4@ZnO materials were successfully obtained with different surface charge characteristics. The samples were then applied in the photodegradation methylene blue (MB) dye under irradiation. It was possible to note that by adding 10 %wt of ZnO boosted photocatalytic degradation of MB dye when compared to pristine ZnWO4. The morphology of ZnO nanoparticles observed on the ZnWO4 had a strong influence on the photocatalytic efficiency of the final ZnWO4@ZnO material, also indicating a synergistic effect between both ZnWO4 and ZnO.

Acknowledgements: UFPB, CNPq/MCTIC, CAPES and FAPESQ/PB-SEECT.

References:

[1] REDDY, Ch. V. et al. Journal of Environmental Management, 268 (2020) 110677.
[2] M. Arellano-Cortaza, M. et al. Ceramics International, 47 (2021) 27469.

Catalytic reduction of 2,4-dinitrophenol with gold nanoparticles

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2,4-dinitrophenol is among the most harmful phenolic derivatives for humans and the environment. It is a toxic organic substance common in industrial effluents and can also be found in aquatic environments near urban and agricultural areas1. The objective of this work was to carry out the degradation through a catalytic reduction using sodium borohydride as reducing agent and gold nanoparticles as catalysts to reduction of 2,4-dinitrophenol to 2,4-diaminophenol, which is less harmful and has important industrial applications such as in the manufacture of dyes and color accelerator in photographic developers2. The colloidal solution of gold nanoparticles synthesized from 2.5×10^{-4} mol L^{-1} citrate was more uniform and homogeneous morphology, with a greater number of particles and better distribution. Regarding to size distribution, this sample showed lower standard deviation in relation to the sample with a higher concentration of citrate, so it was chosen for the reduction of the nitrophenolic compound, showing good degradation efficiency, especially when using a more concentrated solution of nanoparticles.

Acknowledgement: CNPq, FINEP, FAPESP.

References:
1)-ARORA, P. K.; SRIVASTAVA, A.; SINGH, V. P. Bacterial degradation of nitrophenols and their derivatives. Journal of Hazardous Materials, v. 266, p. 42- 59, fev. 2014.
2)- DIN, M. I. et al. Nanocatalytic Assemblies for Catalytic Reduction of Nitrophenols: A Critical Review. Critical reviews in analytical chemistry, v. 50, n. 4, p. 322-338, jul. 2020.

Characterization of TiO_2 nanotubes obtained onTi-29Nb-13Ta-4.6Zr substrates: Mechanical and Compositional Properties for Photocatalytic applications

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TiO_2nanotubes (TNTs) are oxide films grown on Ti alloys substrates through electrochemical anodization and have found important practical applications such as pollutants photodegradation. This derives from their properties as semiconductor with narrow band gap and high surface area, promoting chemical reactions between the TNTs and organic molecules when it is exposed to light. Commercially pure Ti has been the most studied substrate for TNTs formation, with varying electrochemical parameters resulting in different properties and characteristics of the nanotubes [1]. This study aims to investigate the growth, mechanical, and compositional properties of TNTs grown on Ti-29Nb-13Ta-4.6Zr alloy (TNTZ), with a focus on their potential applications in pollutants degradation. After synthesis by anodization, well-ordered and homogeneous nanotube formation was observed by SEM, without residual ribs, which is beneficial for photocatalysis applications, reducing the electron-hole contact. Deconvolution of XPS spectra showed high variability in oxide compositions increasing with film depth, possibly attributed to defects in the crystal lattice due to oxide formation, which in turn lead to different properties of the nanotubes. Moreover, XPS depth profiling showed dominance of high oxidation states on the tube surface, indicating that the nucleation process leads to formation of different morphologies, likely associated with induction of defects in the oxide lattice, forming metastable oxides [2]. After annealing, anatase crystal structure was obtained, which is known to exhibit improved properties for photocatalytic applications. Regarding photodegradation, the use of nanotubes grown on TNTZ increased its efficiency by up to 82% in dye degradation compared to TNTs grown over CP2.

Acknowledgements:

CEM UFABC and CAPES.

References:

[1] Ge, Ming-Zheng, et al. Nanotechnology Reviews 5.1, 75-112. (2016)

[2] Li, Zebiao, et al, Materials Science and Engineering: B, 286 (2022)

Clay minerals and mesoporous ordered silica functionalized with 3-aminopropyltriethoxysilane for adsorption of anionic pollutant species

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High levels of nitrate ions (NO3-) mainly due to the increasing global fertilizers use, and chromate ions (CrO42-) coming from different industries, such as leather tanning and textile industries, in the surface and groundwater, are increasingly becoming a widespread global problem. Thus, the development of effective materials for removal of nitrate and chromate ions from aqueous media is essential. This work aims to prepare ordered mesoporous silica, type SBA-15, and natural clay minerals (kaolinite and vermiculite) functionalized with 3-aminoprylltriethoxysilane (APTES) for adsorption of nitrate and chromate ions from contaminated aqueous solutions. SBA-15 has been widely studied due to its unique properties, such as non-toxicity, chemical stability, structural and textural properties, such as high specific surface area and pore volume, that are interesting for the adsorption of pollutants. Clay minerals also have good adsorption characteristics such as cation exchange capacity, in addition to high surface area, and low cost, which is economically advantageous. Functionalization of SBA-15 and clay minerals materials with amino groups (APTES) was carried out in toluene by grafting method. All prepared materials were characterized by several physicochemical techniques. Small angle X-ray scattering curves showed that the typical mesoporous structure of SBA-15 was preserved after the grafting procedure. Fourier transform infrared spectroscopy and thermogravimetry data of all materials show the presence of APTES, evidencing the functionalization of the materials. Scanning electron microscopy images showed that the particles morphology remains intact after the functionalization and adsorption process. Adsorption experiments were performed to investigate the adsorption of the anions onto SBA-15 and the functionalized clay minerals. The results were promising for the adsorption of anions, with the amino-functionalized SBA-15 showing a higher adsorption performance.

Co and Fe-doped $MoSe_2$ for photocatalytic removingwater pollutants under UV–Light

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Two-dimensional materials have been extensively studied and considered for various applications, such as energy conversion and storage, electronic devices, photocatalytic, and optoelectronic systems. Among them, dichalcogenides compounds (MoS_2, $MoSe_2$, $MoTe_2$) are a particular class of 2D systems that have been studied and applied in several areas of knowledge. Molybdenum diselenide has great potential for the degradation of water pollutants, but its performance is limited by active sites and recombination of photogenerated electron-hole pairs. Also, the catalyst doped with magnetic nanoparticles enables its easy separation from the reaction mixture, thus making its recycling and reusability simple and efficient. In this study, $Mo_{(1-x)}M_xSe_2$ (M=Fe, Co) compounds were synthed by a microwave-assisted solvothermal method. Their structural, microstructural properties are investigated using X-Ray Diffraction, Raman Spectroscopy, UV-vis Spectroscopy, and Field Emission Scanning Electron Microscopy. Photocatalytic dye degradation was analyzed using these samples on rhodamine B (RhB) and methylene blue (MB). Photocatalytic experimental results revealed a better adsorption and photodegradation of rhodamine B (RhB) and methylene blue (MB) were achieved with doped-$MoSe_2$than with pure $MoSe_2$under dark conditions and UV-light irradiation.

Acknowledgments:

Multiuser Central Facilities (CEM/UFABC) and financial support by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES).

References:

[1] Yang, Shu, et al. ACS Applied Nano Materials, 3(3), 2278-2287(2020).

[2] Yang, Xi et al. Applied Surface Science, v. 457, 214-220 (2018).

Comparison of synthesis methodologies for the oxides SiO2-ZrO2 and SiO2-ZrO2-BaO for application as photocatalyst

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Photocatalysis has been widely studied in water treatment through the degradation of organic pollutants, such as dyes. Several metallic oxides have been studied for this purpose, such as zirconium and barium oxides [1], but these oxides have a low surface area, so silica has been used to assign a greater area for these materials [3]. In this work, the mixed oxides SiO_2-ZrO_2(SZ) and SiO_2-ZrO_2-BaO (SZB) were synthed by Pechini's methods (MPE) and metal-chitosan complexation (CMQ). To elucidate the characteristic and textural properties of these materials they were then characterized by different techniques. In the physisorption analyses, using the BET method, a greater surface area was observed for the SZB oxide synthed by the CMQ method (203 m^2/g), while the SZ oxide presented a greater area when synthed by the MPE method (335 m^2/g). By XRD it was observed that the materials were amorphous. In the diffuse reflectance spectroscopy it was observed that only the oxides synthed by CMQ presented band gap energy characteristic of semiconductors, being 2.93 eV for the SZ-CMQ oxide and 3.45 eV for the SZB-CMQ. After the characterizations, the materials were applied in the photodegradation of methylene blue, being irradiated at 273 nm and 365 nm. The reactions were carried out with a total time of 70 min. The SZ-CMQ material showed greater dye photobleaching when irradiated at 365 nm (32% photobleaching), while the SZB-CMQ material had greater photobleaching when irradiated at 273 nm (35.5% photobleaching). For both materials, an equilibrium pattern was not observed in the photocatalytic efficiency curve, so it was necessary to increase the reaction time.

Acknowledgment:
To funding agencies FINEP, CNPq, CAPES, FAPEAL e o INCT-Catálise.

References:
[1] M. Miodyńska and B. Bajorowicz, Solid State Sciences, 74, 13-23. (2017).
[2] B.C.B. Salgado and A. Valentini, Braz. J. Chem. Eng, 36, 1501-1518 (2020).

Composites of titanate de nanotubes modified by points of carbono (C-dots/NaTNTs).: Synthesis, Characterization and Study of Physical Chemical Properties

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The present study reports the development of a composite composed of sodium titanate nanotubes modified by points of carbon (C-dots / NaTNTs), from a source based on titanium oxide and babassu coconut mesocarp, respectively. The composite was successfully synthesized through a hydrothermal procedure. Their structural, microstructural, morphological and optical properties were analyzed by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Ultraviolet and Visible Spectroscopy (UV-Vis), Raman and Fourier Transform Infrared (FTIR).The characterization results indicated the formation of sodium titanate nanotubes, carbon dots and the successful deposition of C-dots on NaTNTs. As intended, the hybrid nanomaterials showed an increase in the energy absorption range compared to pure NaTNTs. In this sense, this study presents an available method to modify titanate nanomaterials, and the new C-dots/NaTNTs hybrid material developed may be promising for photocatalytic applications aimed at removing organic pollutants that cause environmental impacts.

References.:

1. B. C. Viana, O. P. Ferreira, A. G. Souza Filho, J. Mendes Filho, and O. L. Alves, Journal of the Brazilian Chemical Society 20, 167 (2009).
2. T. Kasuga, M. Hiramatsu, A. Hoson, T. Sekino, and K. Niihara, Advanced Materials 11, 1307 (1999).
3. R. S. Costa, M. O. De Castro, H. Gabriela, F. D. S. Delite, M. Strauss, O. P. Ferreira, D. S. T. Martinez, and B. C. Viana, Carbon Trends 5, 100133 (2021).

Controllable solvothermal synthesis and morphology-dependent photocatalytic properties of ZnO nanostructures.

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Pollution caused by improper disposal of different pollutants, such as organic dyes and pharmaceutical drugs, brings the search for new technologies for the decontamination of water bodies. Several methodologies, especially heterogeneous photocatalysis have proven to be used to degrade these pollutants in water [1,2]. Among different photocatalytic oxides,ZnO needs to be highlighted for presenting intrinsic carrier concentration depeding on the particle morphology that can strongly influence the photocatalytic properties [3]. Thus, the present work aims to investigate the photocatalytic efficiency of different ZnO nanostructures obtained by the microwave-assisted solvothermal method in different synthesis media at 100°C for 1h. The samples were characterized by X-ray diffraction (XRD), infrared spectroscopy (IR), UV-Vis spectroscopy, scanning electron microscopy (FE-SEM) with energy dispersive X-ray spectroscopy (EDS), electron paramagnetic resonance (EPR)and transmission electron microscopy (TEM). Our results demonstrated that all samples presented well-crystallized particles with different and shape as well as distinct growth orientation and charge carrier concentration. From the photocatalysis data, it was possible to observe that the flower-like ZnO nanostructures presented a photodegradation efficiencyof 70% of methylene blue dye for 4 h, while the star-like and sphere-like ZnO nanostructures showed an efficiency of 45% and 15%, respectively.

Acknowledgements: UFPB, CNPq/MCTIC, CAPES and FAPESQ/PB-SEECT.

References:

[1] Catanho, M., Malpass, G.R.P., Motheo, A.D.J. Química Nova, 29 (2006) 983.
[2] Rajbonhshi, B.M., Handbook of Smart Photocatalytic Materials, Elsevier, 2020, p. 127.
[3] M. Arellano-Cortaza, M. et al. Ceramics International, 47 (2021) 27469.

Degradation of the azo Remazol Red RB dye usingsilver nanospheres

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Water is an extremely necessary asset for the maintenance of human life in different spheres, however, access to drinking water is still a rare item for millions of people. With globalization and industrial expansion, environmental pollution become more evident, as can be seen in the aquatic environment, for example[1]. One of the most common forms of effluent contamination is through improper disposal of dyes from cosmetic, pharmaceutical, and textile industries, the latter being one of the biggest contributors to this problem. These dyes are organic substances that mostly have the azo group in their structure (-N=N-), which are capable of giving color to a substrate through chemical affinity. These substanceshave carcinogenic, mutagenic, and genotoxic potential, and methods that contribute to thecomplete degradation of these species are still needed. Catalytic methodologies for this degradation process are viewed positively. The literature describes some methods for thispurpose, such as nanocatalysis, in which the use of nanoparticles, specifically silver nanoparticles (AgNPs) have been highlighted [2]. In this work, AgNPs were synthesized with3 stabilizers, ascorbic acid (AA), polyvinyl alcohol (PVA), and sodium citrate, and were applied in the degradation of the azo dye, remazol red RB. The optical and structuralcharacterizations confirmed the formation of stable nanospheres. The degradation of the azo dye with the catalysis was evaluated via UV-Vis in a maximum time of 30 minutes. Ourresults showed that these AgNPs were efficient catalysts for the degradation of the organicdye in an aqueous medium, with a faster, cheaper, and simpler method than those alreadydescribed in the literature.

Acknowledgments: The authors thank the UFPE, CNPq, FACEPE (IBPG-1107-1.06/22), and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020).

References:
[1] AL-DEGS et al. Water Res., 2000, 34, 927
[2]HANAFI and SAPAWE, Materials Today:Proceedings,2020,31,A141.

Development of Nanoadsorbent Based on MCM-41 and ZIF-8, for Application in the Removal of Organic Pollutants

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In view of the scenario of environmental pollution, the use of the Adsorption-Process, as a technology for removing contaminants, stands out especially because it is low cost, it allows the recovery of the adsorbent and also due to the high variety of adsorbent materials [1], among them we highlight the porous materials, giving emphasis to the mesoporous silica MCM-41 (Mobil Crystalline Materials) and the Zeolitic Imidazolate Framework-8 (ZIF-8). Both materials have a surface area with specific adsorption sites, selectivity, porosity, favorable adsorption kinetics, and renewability [2,3]. The present work aims to obtain a heterostructure from the synthesis by in situ coprecipitation of ZIF-8 on the surface of MCM-41, improving its individual characteristics, and thus, developing a promising functional material to be used in adsorption. This material was characterized by several physical-chemical techniques, such as XRD, FTIR, N2 adsorption (BET analysis), showing an interaction between the pristine materials while maintaining their structures and textural properties.

Acknowledgment: IFMA, BIONANOS - UFMA.

References

1. Y.S. Ho, Review of second-order models for adsorption systems, J. Hazard.Mater. 136 (2006) 681-689. doi:10.1016/j.jhazmat.2005.12.043.
2. ZHOU, L.; LI, N.; OWENS, G.; CHEN, Z. Simultaneous removal of mixed contaminants, copper and norfloxacin, from aqueous solution by ZIF-8. Chemical Engineering Journal, v. 362, p. 628-637, Apr. 2019. DOI. 10.1016/j.cej.2019.01.068.
3. XIE, Y.; ZANG, Y.; OUYANG, J.; YANG, H. Mesoporous material Al-MCM-41 from natural halloysite. Phys Chem Minerals, v. 41 p. 497-503, 2014.

Effect of Bi_2WO_6 doping with Zn^{2+} on the photodegradation of methylene blue irradiated underUV light.

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Abstract

The accentuated population growth in the 20th century enabled large-scale industrial production characterized by the intense use of synthetic chemical compounds such as insecticides, organic dyes, drugs, among others. The waste generated by these activities is usually discarded in the ecosystem, leading to the accumulation of a large number of pollutants in natural water bodies and, in turn, causing significant damage to the environment and human life. Therefore, in order to contain the deleterious effects of organic pollutants on water bodies, it is essential to develop materials with good photocatalytic activity, such as tungstates [1]. In this context, the present study aims to obtain pure Bi_2WO_6 nanoparticles and Bi_2WO_6 nanoparticles doped with 4% Zn^{2+} through the sonochemical method. The photocatalytic activity of the pure and doped samples was verified from the degradation of methylene blue dye (MB). The XRD results showed the presence of a single orthorhombic phase and it was verified that the doping was effective, since there was no formation of a deleterious phase. The micrographs performed by FEG-SEM of pure and doped samples showed rod-shaped and spheroidal structures. According to the UV-vis analysis, the doped sample showed the highest redshift at the absorption edge compared to the pure sample. The pure sample had the best performance in the degradation of the methylene blue dye compared to the doped sample, when subjected to UV irradiation, with a percentage of dye degradation in the presence of Bi_2WO_4-pure exceeding 80%.

Acknowledgements: To UFRN, LSQM, PPGCEM and CAPES.

References:

[1] Silva JMP, Andrade Neto NF, Teodoro MD, Paiva AEM, Bomio MRD, Motta F V. Fabrication of new Ag_2CrO_4/Ag_2MoO_4 and Ag_2CrO_4/Ag_2WO_4 heterostructures with enhanced degradation of the mixture of dyes by photocatalysis. J Alloys Compd 2022;928:167136. https://doi.org/10.1016/j.jallcom.2022.167136.

Effect of dopant and sintering aid on solid-state reactive sintering of $BaCe_{0.2}Zr_{0.7}M_{0.1}O_{3-\delta}$ perovskite (M=Y, Gd, Sm, Yb) as electrolyte for protonic ceramicfuel cells

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Solid Oxide Fuel Cells (SOFCs) with an oxide-ion-conducing electrolyte are efficient, clean, and sustainable energy-conversion devices. However, the high performance of SOFCs occurs at a high temperature (≥ 800 °C), which can cause material degradation and increase balance-of-plant costs. Proton-conducting SOFCs (Protonic Ceramic Fuel Cells, PCFCs) alleviate this problem on operating at lower temperature (600-800 °C) since the activation energy for conduction of protons is lower than that of oxide ions. Perovskite proton conductors $BaCe_xZr_{1-x}O_{3-\delta}$are very promising but their refractory nature means that densifiying electrolytes requires high temperatures and long sintering times. To reduce the temperature and time of conventional sintering, alternatives have been explored, such as solid-state reactive sintering using sintering aids [1]. In this work, we investigated the effect of the dopant and ZnO sintering aid in the system $BaCe_{0.2}Zr_{0.7}M_{0.1}O_{3-\delta}$(M=Y, Gd, Sm, Yb) on solid-state reactive sintering and densification. Stoichiometric powder mixtures were prepared with and without the addition of 2 mol% ZnO as sintering aid. Sm- and Gd-doped samples with the addition of ZnO sintered to >90% relative density with one heat treatment at 1650 °C for 4 hours from the compacted starting powders. Whereas other dopants and samples without ZnO did not densify. The total conductivity determined by impedance spectroscopy for the sample $BaCe_{0.2}Zr_{0.7}Sm_{0.1}O_{3-\delta}$with 2 mol% ZnO was 2.60 mS. cm^{-1}at 600 °C in air, a value higher than that of the analogue obtained via the conventional ceramic routewith multiple calcination and milling steps.

Acknowledgements:

The authors gratefully acknowledge CAPES-Brazil, CNPq-Brazil, ICV-CSIC-Spain, and C-LABMU-UEPG.

References:

[1] Medvedev, D. A. et al. Formation of dense electrolytes based on $BaCeO_3$and $BaZrO_3$for application in solid oxide fuel cells: the role of solid-state reactive sintering. Rev. J. Chem., v. 5, p. 193-214, 2015.

Effect of Li or Mg ionic doping on photoelectrochemical behavior of CuWO4photoelectrodes

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Solar water splitting is an alternative method to produce H2 using sunlight and specialized semiconductors [1]. Ionic doping in semiconductors is considered efficient strategy to enhance the charge transport and avoiding its rapid recombination. This study, a simple polymeric precursor method was employed for the fabrication of pristine copper tungstate (CuWO4) film and that of varying doping concentration (1;3;5%) of lithium (Li:CuWO4) and magnesium (Mg:CuWO4). Optical, morphological, and structural properties of the synthed materials were characterized using diffuse reflectance spectroscopy (DRS), emission-scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), respectively. Spherical shaped nanostructures were observed, just like confirmed triclinic structure and the presence of dopants with elongation of crystalline lattice [2]. The impurities occupied interstitial sites in the host CuWO4 lattice and induced shift for lower optical band gap energy [3]. The presence of dopants was clearly observed with photoelectrochemical response, modifying n-type behavior of host film for p-type in the doped CuWO4 films. Doping with Li or Mg in host CuWO4 electrodes favored the inhibition of the charge recombination. Moreover, an excellent material for photoelectrochemical application.

References

[1] S. Wrede, H. Tian. Towards sustainable and efficient p-type metal oxide semiconductor materials in dye-sensitized photocathodes for solar energy conversion. Phys. Chem. Chem. Phys., 22, (2020), pp. 13850-13861.

[2] S. Aksoy, O. Polat, K. Gorgun, Y. Caglar, M. Caglar. Li doped ZnO based DSSC: Characterization and preparation of nanopowders and electrical performance of its DSSC. Physica E 121 (2020) 114127.

[3] C. Belkhaoui, N. Mzabi, H. Smaoui, P. Daniel, Enhancing the structural, optical and electrical properties of ZnO nanopowders through (Al + Mn) doping, Results Phys. 12 (2019) 1686-1696.

Electro-spun membrane for photodegradation of emerging pollutant

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In recent years there has been an increase in the development of electrospinning materials due to the functional characteristics obtained through a quickly and simply way [1]. This work successfully prepared electrospinning particles of polyvinylpyrrolidone containing TiO2 synthesized by the Pechini method. Different types of TiO2 annealed at 400 and 500 °C were incorporated into a polyvinylpyrrolidone polymer resin and the photocatalytic activity of membranes functionalized with TiO2 was demonstrated with the degradation of venlafaxine. The membranes were extensively characterized using techniques such as attenuated total reflectance, Fourier transforms infrared spectroscopy, scanning electron microscopy and oxide particles by X-ray diffraction, UV-visible absorption and the Brunauer-Emmett-Teller adsorption method to correlate the characteristics of oxide particles with their performance. The results of photodegradation with venlafaxine in sodium phosphate buffer monitored by voltammetry showed excellent photocatalytic activity of TiO2 particles immobilized on the membrane structure. After 90 min of UV irradiation, a degradation of up to 85% of the drug was observed. The lowest annealing temperature (400 °C) produced a TiO2 phase mixture rich in anatase on the PT 400 C membrane with increased surface area, generating the adsorption sites of the venlafaxine molecule. A reuse test of the functional materials confirmed that the TiO2 particles were successfully incorporated into the electro-spun membrane and revealed that organic matter photodegradation is possible. Acknowledgments: this work was carried out with the support of the Coordination for the Improvement of Higher Education Personnel-Brazil (CAPES) and CNPQ. References:[1] Kumar, P.; Bharti, R. P.; Kumar, V.; Kundu, P.P. Polymer Electrolyte Membranes for Microbial Fuel Cells: Part A. Nafion-Based Membranes. In Progress and Recent Trends in Microbial Fuel Cell Elsevier B.V.: Índia, 2018; pp. 73-85

Elevation of photocatalytic activity with the mixture of anatase and rutile phases in TiO_2 nanotubular structures

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Persistent pollutants such as dye residues from the textile industry, drugs and even microplastics cannot be retained or eliminated with conventional methods of effluent treatment, requiring the use of more specific and advanced techniques for these treatments. Heterogeneous photocatalysis has shown efficiency for this purpose. It consists of the use ofa semiconductor material that, when irradiated with light at specific energy, initiates a series of electronic reactions, generating highly oxidative species in solution, such as hydroxyl radicals. The present work investigated the photocatalytic efficiency of titanium dioxide nanotubular structures in its anatase phase and in proportions with 27% of the rutile phase, for the photodegradation and minieralization of methylene blue dye. Nanotubular titanium dioxide plates measuring approximately 1.3 cm2 were kept in contact with the dye solution at 15 mg/L, in a quartz reactor, at 25ºC, with different irradiation times using a solar simulator. The reaction product was analyzed by UV-Vis and TOC, relating the decrease in the intensity of the bands with the reduction in the dye concentration in solution and, consequently, its photodegradation. The reduction in TOC concentration was attributed to dye mineralization. In 6 h of irradiation, 87% of discoloration of the solution was reached, and 37% of removal of OC, using the material with 27% of rutile in mixture of phases. Which represents an increase in efficiency by approximately 40% compared to the material with the pure anatase phase. Thus, better results are expected with optimizations related to the of the anodized area, light energy used in photocatalytic tests or even increased exposure time.

Emergent contaminants adsorption from aqueousmedia by biochar/bentonite composite

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The lack of using wastewater treatment processes in Peru let some drugs, such as oxytetracycline (OTC), increasing its concentration in water bodies year by year; for this reason, they are considered a risk associated with wastewater discharges into the aquatic environment. (Nieto-Juárez et al., 2021). Therefore, some advanced treatments are necessary to remove that contaminant. Among the removal methods, adsorption is an efficient method due to the feasible preparation of biochars from inexpensive materials, giving a large surface area material. The aim of this work is to adsorp OTC from water samples by a composite comprising 2 active materials: natural bentonite and biochars obtained from reed wood (Phragmites Australis). The preparation process was as follows: pretreated reed wood (cut up, blended and rinsed) and bentonite (rinsed and activated with 2.0 M HCl solution) in a1:1 ratio was mixed with ZnCl2 solution and stirred for 10h. After dried, a pyrolytic process was carried out at 400 °C in N2 atmosphere for 4 h at 10 °C min-1. Diffraction patterns revealed the presence of quartz, montmorillonite, and feldspar structures, being the crystalline face (001) of montmorillonite the main peak. FTIR spectra showed the stretching of de C=C bond in the aromatic ring at 1587 cm-1 and 2 bands at 1022 cm-1 and 794 cm-1 corresponding to bentonite. BET isotherm gave a surface area of 515 m2.g-1. Kinetic studies showed that the composite was able to adsorb upon 87.4 % of OTC from 20 ppm solutions in 90 min with a dosage of 50 mg/100 mL. The Temkin isotherm best fit the results in equilibrium state. Results suggested that the material could be used to effectively remove OTC from aqueous solutions.

Reference

Nieto-Juárez, J. I., Torres-Palma, R. A., Botero-Coy, A. M., am Hernández, F. (2021). Pharmaceuticals andenvironmental risk assessment in municipal wastewater treatment plants and rivers from Peru. EnvironmentInternational, 155.

Evaluation of a Photoelectrochemical Approach Basedon Iron Vanadate Electrode Toward Immunosensing Detection of SARS-CoV-2 Glycoprotein

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This study describes a straightforward and affordable approach to constructing a photoelectrochemical (PEC) platform to detect SARS-CoV-2 glycoprotein using an iron vanadate semiconductor. The semiconductor was obtained via the Successive Ionic Adsorption and Reaction process on indium-doped tin oxide substrate (ITO). Its structure and morphology were thoroughly characterized.[1,2] To assess the interaction between SARS-CoV-2 and anti-SARS-CoV-2, the immunosensor surface was incubated with a SARS-CoV-2 solution. Amperograms were recorded by applying different potentials (+0.4 and +0.5 V vs. Ag/AgCl) in the absence and presence of visible light in phosphate-buffered saline (0.1 mol L^{-1}, pH 7.01). Formic acid (1 mmol L^{-1}) was also evaluated as a suitable sacrificial agent able to scavenge photo holes. The optimized anti-SARS-CoV-2-FeVO/ITO PEC platform was modified with 5 μg mL^{-1}antibody to detect SARS-CoV-2 glycoprotein. A decrease in photocurrent was observed as the concentration of SARS-CoV-2 increased from 1 fg $mL^{-1}$to 460 pg mL^{-1}. After ten successive essays, the RSD was calculated and found to be 3.2 and 3.4% for 60 and 260 pg mL^{-1}SARS-CoV-2 solutions, respectively. Furthermore, the biosensor could differentiate serum obtained from COVID-19 positive versus negative clinical samples, indicating that this platform holds promise as a rapid, efficient, and reliable method for detecting SARS-CoV-2 protein.

Acknowledgment:
CAPES, LMEM, INCT-Bio, and ESPEC.

References:
[1] M. R. S. Pelissari, L. P. Camargo, et al., J. Mater. Sci., 57, 7173-7190 (2022).
[2] M. M. Sajid, H. Zhai, et al., Mater. Sci. Semicond. Process., 129, 105785 (2021).

Evaluation of the photocatalytic properties of $ZnWO_4$ in the degradation of methylene blue

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Zinc Tungstate ($ZnWO_4$) was synthed by the Pechini method. To optimize the synthesis of ZnWO4, precursors such as Zinc Acetate dihydrate $Zn(NO_3)2.6H_2O$ and Zinc Nitrate hexahydrate ($C_4H_6O_4Zn.2H_2O$) were tested, in addition to calcination levels of 400°C/2h followed by 700°C/4h and direct plateau of 700°C/4h. This second conditions and the use of $Zn(NO_3)2.6H_2O$ as precursor resulted in the synthesis of singe phase $ZnWO_4$, which was confirmed by characterizations by X-Ray Diffraction, Infrared (IR) Spectroscopy and Raman Spectroscopy in order to confirm the formation of $ZnWO_4$by the Pechini method. From the Absorption Spectroscopy in the UV-vis region, the band gap of $ZnWO_4$was calculated by the Wood and Tauc method and a value of 3.20 eV was obtained. Morphological analysis by scanning electron microscopy showed the formation of agglomerated particles. The zero charge point (ZCP) for ZnWO4 was at pH = 2.19. As pH values increase, the semiconductor has a negative surface. The evaluation of photocatalytic activities of zinc tungstate was carried out under UVC irradiation (λ= 254 nm) for terephthalic acid (AT) hydroxilation for evaluation of hydroxyl radicals ($^{\cdot}OH$) by fluorescence spectroscopy. The photodegradation of methylene blue dye (MB), using concentrations of 0.5, 0.8 and 0.85 mg mL-1 was also evaluated and the effect of pH on the photocatalytic activity was verified, considering pH = 5, 7 and 11. $ZnWO_4$had an efficiency of 99.3% in MB discoloration after 2h of irradiation. The meaningful formation of OH radicals and the MB adsorption, which depends of the material's surface charge, were determining factors for $ZnWO_4$to achieve a better photocatalytic performance in the degradation of the MB cationic dye.

Acknowledgments: CAPES/MEC, PRONEX/FAPESQ-PB/CNPq-MCTI, CT-INFRA/FINEP/MCTI

References:

[1] Alkaim, A. F. *et al.* Asian J. Chem. 26, 8445-8448 (2014).
[2] Žerjav, G., Albreht, A., Vovk, I. & Pintar, A. Appl. Catal. A Gen. 598, 117566 (2020).

Evaluation of the potential use of recycled materials for adsorption in treatment

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The search for sustainable and economically feasible materials for wastewater treatment has been an important line of research in materials engineering. In this context, the use of recycled materials as adsorbents has been a promising alternative, since these materials have adsorbent properties and can be obtained from solid waste, thus reducing the environmental impacts. In this work, we evaluated the potential use of different recycled materials, such as plastics and ceramic materials, for the adsorption of contaminants in effluents. Characterization studies of the materials, adsorption isotherm analyses, and pilot scale tests were performed. The results indicated that the recycled materials present adsorption capacity comparable to conventional materials used in wastewater treatment.The results obtained indicate that the use of recycled materials as adsorbents can be a promising alternative for effluent treatment, contributing to the reduction of environmental impacts and the development of sustainable materials.

Fast synthesis of NaNbO3 nanoparticles with high photocatalytic activity

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Research using semiconductors as photocatalysts has been carried out since the 70s. In this photocatalytic process, photons with energy equal to or greater than the band gap energy absorbed by the semiconductor, generate electron/hole pairs, thus creating oxidizing and reducing sites in its surface. These sites can degrade various pollutants into effluents, generate H2 and also reduce harmful gases. Many factors affect the activity of photocatalysts such as particle size, due to the surface/volume ratio, making nanoparticles very promising. Sodium niobate (NaNbO3) is a promising photocatalyst however, the natural shape of particles are large cubes, but research has already reported nanoparticles with high photo-catalytic activity [1]. Therefore, the objective of this work was to obtain NaNbO3 nanoparticles in a shorter time and apply different heat treatment temperatures in order to analyze the influence on the photocatalytic activity. The samples ob-tained had their photocatalytic activity measured through the photodegradation of Rhodamine B (RhB),a dye widely used in industries and known for its high tox-icity, good stability, and nonbiodegradability. In this study, we obtained NaNbO3 nanoparticles in the shortest time ever reported in the literature and demonstrated that with a simple thermal treatment it is possible to change the shape of the par-ticles, from nanowires to nanograins. All samples showed high photocatalytic ac-tivity, especially the nanograins, due to their smaller particle size and higher crys-tallinity. Reuse tests demonstrated the stability of the photocatalysts. Therefore, this study contributes with information for obtaining highly favorable NaNbO3 na-noparticles for photocatalytic applications.Acknowledgements: CAPES, CNPq, FAPERGS, UFPEL, UNESP Campus Bauru, UNIFESP Campus São José dos Campos, UNIPAMPA Campus Bagé and Thissiana Fernandes.References:[1] D. Fernandes et. al., Ceramics International vol. 47, 10185 (2021).

Fe-birnessite synthesis from Amazon mining waste and their photocatalytic potential

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Recovery of mining waste could be a significant alternative to solve the issue of tailings in the Brazilian Amazon rainforest. In this study, manganese (Mn) oxide tailings were collected from the Azul manganese mine in Pará state, located in the northern region of the Carajás Mineral Province. The material underwent thermal treatment, resulting in the production of sodium-layered octahedral manganese oxide (Na-OL-1), which was then subjected to an ion exchange process using an iron precursor (FeCl) to obtain Fe-birnessite (Fe-OL-1). DRX, EDS, and Raman analyses confirmed the successful substitution of iron in the lamellar structure. Fe-OL-1 was used as a catalyst in the treatment of 50 mg/L of p-nitrophenol (PNP) in batch mode using heterogeneous dark-Fenton and photo-Fenton processes with hydrogen peroxide as an oxidant. Both processes achieved a PNP removal efficiency above 90% after 60 minutes of treatment, but the use of simulated sunlight (photo-Fenton) led to a mineralisation rate of 62%, compared to ~40% without light. At the end of both treatments, the sample was determined to be non-toxic based on an artemia salina toxicity assay. **Acknowledgements**: This work was supported by FNDCT through Finep [process 0122056900]. VNL, CEML, CISF and TSA are grateful to CNPq for their PCI fellowship. Cetene is a member of Sistema Nacional de Laboratórios em Nanotecnologias (SisNANO) of MCTI.

Fe-TiO_2/vermiculite for photocatalytic application

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The immobilization of TiO_2on vermiculite clay mineral (Verm) is a strategy to improve the efficiency of the photocatalyst [1]. Doping the semiconductor with metallic ions, such as Fe^{3+}, is an alternative to modify the optical properties of the material and enable its excitation in regions of lower energies [2]. The present work aimed to evaluate the photocatalytic properties of TiO_2/Verm. The material was synthed via sol-gel and then calcined at 400°. XRD, diffuse reflectance, and SEM characterized the composite. The photocatalytic tests were carried out against methylene blue (30 ppm) using a 160w mercury vapor lamp as a source of Vis radiation. Dye discoloration was evaluated from the dye absorption band at 663nm. From the diffractograms, vermiculite, and TiO_2in anatase are identified form. From the reflectance data, the presence of Fe^{3+}caused a decrease in the material''s band gap (From 3.2 to 2.95 eV). There is no significant change in material morphology compared to Verm. The results of the photocatalytic testicles appreciated that TiO2 promoted 6% discoloration of the MB (1.8 ppm). While Fe2.5-TiO_2/Verm promoted 49.9% of discoloration after 120 min, being 3.31 ppm from photobleaching and 11.66 ppm by adsorption (in the dark). The results demonstrate significant advances in developing photocatalysts for environmental remediation applications.

Functional carbon-based catalyst derived from coffee grounds for efficient degradation of organic dyes via sono-fenton process

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The contamination of water bodies by effluents and its subsequent environmental impacts have led to a growing need to develop effective water treatment strategies. Advanced oxidative processes (AOPs) have gained widespread recognition for their ability to mineralize non-biodegradable molecules. Among the AOPs, Fenton processes have been extensively studied for their in-situ formation of reactive oxygen species (ROS) in the presence of Fe2+ ions. However, the classic Fenton process has several limitations, including a restricted pH range, catalyst recovery, and reagent volume. In this context, the ultrasound-assisted Fenton process (Sono-Fenton) has emerged as a promising alternative due to its ability to optimize degradation time and increase ROS formation [1]. This study aimed to prepare, characterize, and apply a carbon-based catalyst obtained from coffee grounds and loaded with FeCl3. X-ray diffraction analysis confirmed the deposition of FeCl3 on the carbon support and the infrared data confirmed the presence of peaks characteristic of -OH stretching of alcohols, C=O/aromatic and C=C of aromatic rings. SEM micrographs depicted a porous structure, and EDS data confirmed the uniform distribution of iron along the sample surface. Catalytic tests were performed at room temperature (25°C ± 2) with solutions of methylene blue (MB), rhodamine-B (RhB), and methyl orange (OM) at pH 7, adjusted by using HCl and NaOH 1M. Adding H2O2 marked the beginning of ultrasonic irradiation for all catalytic tests performed. The results demonstrated an impressive efficiency of 95 %, 97 %, and 97% for MB, RhB, and OM within 45 minutes.

Acknowledgments:
The authors gratefully thank CNPq and Capes for the scholarships granted. This work was supported by CNPq (Grants no. 405364/2021-0 and 314590/2021-8).

References:
[1] RAMÍREZ, C. G., CHICA, E., CLEMENTE, A. R. Coupling of Advanced Oxidation Technologies and Biochar for the Removal of Dyes in Water. Water, v. 14, p. 2531, 2022.

g-C3N4 hybrid with meso-metalloporphyrins derived from cardanol for heterogeneous catalytic reduction of4-nitrophenol

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Graphitic carbon nitride (g-C3N4) is a conjugated polymer mainly formed by carbon and nitrogen atoms in a graphite-like structure. It is widely used in photocatalytic reactions, such as in lithium batteries, sensors, and pollutant degradation. However, its catalytic potential is limited due to its small surface area, numerous electron pairs, structural gaps and low UV absorption. Heterojunction via π-π interaction with porphyrin-like macromolecules will increase the surface area, creating more active sites for adsorption. Therefore, in this work porphyrins were derived from cardanol, the main constituent of cashew nut shell liquid (CNDL). They are macrocyclic compounds formed by four pyrrolic rings joined by methynic bonds, with substituents in the meso and/or β-pyrrolic positions. Their internal cavity allows the addition of substituents and metal ions, making them good options to act in catalytic reduction reactions. Hybridization of meso-metalloporphyrins with g-C3N4 may be an alternative for more effective conversion of 4-nitrophenol, a nitrophenolic compound found in wastewater harmful to fauna, flora, and human health, into a less toxic compound, 4-aminophenol, through heterogeneous catalysis. This work describes the synthesis and characterization of nanocomposite g-C3N4 hybrid with meso-metalloporphyrin (Pd ZnP) derived from cardanol, with potential application in the heterogeneous catalytic reduction of 4-nitrophenol in aqueous solutions. The materials were characterized by UV-vis, FT-IR, RMN 1H, TGA, SEM, and X-ray techniques. The kinetic study of the 4-nitrophenol reduction reaction showed reduced reaction time, following a first-order model, proving the efficiency of the synthesis and catalytic result of the new hybrid compounds, g-C3N4 /PdP and g-C3N4 /ZnP. Furthermore, the study demonstrated that the hybrid compounds can be reused after recycling, contributing to the use of catalysts from renewable sources, thereby reducing the environmental impact.

GQD/TiO₂ nanocomposite preparation for the incrementof photocatalytic activity in paracetamol degradation

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In recent years, the use of nanoparticles in the degradation of organic pollutants has gained popularity. Among the numerous photocatalytic materials investigated, titanium dioxide (TiO_2) effectively degrades various pollutants, including pharmaceuticals such as paracetamol. However, the use of TiO_2 in photocatalysis has been limited by its inefficiency and short electron lifetime. Graphene quantum dots (GQDs) have shown great potential in enhancing the photocatalytic activity of TiO_2. The aim of this work is the preparation and facile dispersion of GQDs over TiO_2 to obtain a nanocomposite for paracetamol photodegradation [1]. GQDs were obtained by the commercial graphene oxide 1% dispersion (Williamblythe) with 1.2 mL ammonium hydroxide. The mixture was kept at 180°C for 12 h under constant stirring of 600 rpm. Then, the solution was centrifuged, and impregnatated onto titanium dioxide (TiO_2) prepared by sol-gel methods. The paracetamol degradation efficiency of the GQDs/TiO_2 nanocomposite was evaluated under UV-light irradiation (black light with λ=365 nm). The results revealed that the GQDs/TiO_2 nanocomposite showed 70% paracetamol degradation efficiency in 60 minutes, while the TiO_2 had only 50% efficiency in the same period.

In conclusion, the facile dispersion of GQDs over TiO_2 creates a nanocomposite material with superior photocatalytic activity for paracetamol degradation. It can attribute to GQDs capacity to reduce the recombination rate of photogenerated charge carriers. Therefore, this study broadens the application of GQDs/TiO_2 in photocatalysis and provides a potential solution for removing paracetamol from the aqueous solution.

Acknowledgments: FAPESP (Processo 2019/13471-8).

[1] X. Cao, G. Tian, Y. Chen, J. Zhou, W. Zhou, C. Tian, H. Fu, Hierarchical composites of TiO_2 nanowire arrays on reduced graphene oxide nanosheets with enhanced photocatalytic hydrogen evolution performance, Journal of Materials Chemistry A, 2 (2014) 4366-4374.

GREEN SYNTHESIS OF ZNO NANOSTRUCTURES DOPED WITH LA USING MANGIFERA GUM INDICA ASGROWTH STABILIZER: PHOTOCATALYTIC PROPERTIES.

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ZnO is a highly researched semiconductor because it has interesting properties and many applications [1]. Thus, the present work presents the development through the green synthesis of zinc oxide (ZnO) nanostructures doped with lanthanum (La) stabilized with 1% of Mangifera Indica (MI) gum aiming at photocatalytic applications. There is little research on the use of MI acting as a stabilizer in the doping of ZnO with rare earths, in addition to the inclusion of natural materials in the synthesis can produce products that are less toxic to the environment. To perform the synthesis, the gum of the MI was first extracted using the exudate. Then the synthesis was performed with 1% MI gum, zinc nitrate and lanthanum nitrate that were heated in constant agitation, then it was dried and calcined. For the structural investigation, X-ray diffraction (XRD) was performed and to observe the efficiency of the nanoparticles in the degradation of dyes, the photocatalytic test with methylene blue was performed. In the XRD it was observed that the method used was efficient in the production of ZnO nanoparticles, both for the pure sample and for the woolly doped. A peak attributed to La was observed in the doped nanoparticle that was related to the formation of La_2O_3[2] because of its concentration, it was also observed a deviation of the position of the peaks of the doped sample in relation to pure, as well as a variation of the average of the crystallite. On the photocatalytic activity of the doped material was observed a degradation of 96% of methylene blue in a time of 90 min on UV radiation.

ACKNOWLEDGEMENTS :
To UFPI and UFRPE-UACSA.

REFERENCES

[1] PASCARIU, et al. Preparation of La doped ZnO ceramic nanostructures by electrospinning-calcination method: Effect of La3+ doping on optical and photocatalytic properties. 2019; 476: 16-27.

[2] NGUYEN, LTT, et al. Preparation, Characterization and Photocatalytic Activity of La-Doped Zinc Oxide Nanoparticles. Materials. 2019.

Hydrogen evolution performance of tungsten and molybdenum disulfides-based electrodes

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Hydrogen can be used as an alternative to fossil fuels in energy production. Traditionally, H_2 manufacturing is achieved through the combustion of fossil fuels, yet it results in damageto the environment. H_2 is not naturally available in the environment, and electrochemical methods to split molecules that contain it can be applied. Electrocatalysts that possess botha high current density and low overpotential are critical for successful Hydrogen Evolution Reaction (HER) [1]. Noble metals like Pt and Pt-based materials have been widely applied for HER [2]. The costly expensive of Pt has been encouraged scientists to research and develop new electrocatalysts materials. In this work, two sulfides were investigated to allow the HER in acid media, WS_2 and MoS_2. Transparent glasses coated with fluorine-doped tin oxide were cut into 1cm x 2cm and were used as substrate to deposit the layers of each material. A solution with 4:1 proportion of distilled water and ethanol was prepared and mixed with Nafion 5 wt.% binder. Drop casting technique was applied to coat the samples with 10µl of the WS_2 and MoS_2 solutions until the complete dry at room temperature, for twice. Electrochemical analysis was performed in acid media to determine the overpotential level of each electrode. The results showed that at 350mV the WS_2 electrode started the HER [3], exhibiting better result compared to MoS_2 (500mV). To future investigations, characterizations morphology, electrochemical and spectroscopic of these materials will be realized.

Acknowledgments: We would like to acknowledge the Brazilian foundations: CAPES, CNPq, FINEP and FAPERJ for the funding support for this research, and for the undergraduate and graduated scholarships.

References:

[1] S. Anandhakumar and B. Mauro, Journal of the Brazilian Chemical Society v. 29, p. 226-231(2018).
[2] B. Dongyeon et al., Current Applied Physics v. 30, p. 20-26(2021).
[3] Do, Ha Huu et al., Nano Convergence v. 8, n. 1(2021).

Impregnation of Nb2O5 in loofah sponge for use in heterogeneous photocatalysis

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Heterogeneous photocatalysis is a technique that has been widely used in the degradation of organic pollutants mainly found in effluents from industries [1]. $Nb_2O_5$is a semiconductor material used in several technologies, due to its interesting chemical and physical properties, with good thermal stability and wide bandgap [2]. In photocatalysis, Nb_2O_5has been studied and improved to find greater efficiencies in the degradation of organic dyes [3]. Even though it is a material that presents good results, the idea of impregnating Nb_2O_5in photocatalytic supports can help in greater degradation efficiencies. One of the methods for producing this material is microwave-assisted hydrothermal synthesis. Thus, this work shows the impregnation of Nb_2O_5in loofah sponge through the microwave-assisted hydrothermal synthesis method, to use the material as photocatalytic support in RhodamineB dye degradation processes. The Nb_2O_5impregnated has the characteristic of presentinglow crystallinity of the pseudohexagonal phase, which is predominant for materials not submitted to heat treatment. The SEM images show that there is an impregnation of Nb_2O_5on the surface of the loofah. Preliminary photocatalysis results indicate that the support of vegetable loofah impregnated with Nb_2O_5presents good efficiency of Rhodamine B degradation, being able to degrade 100% of the dye in some minutes of the photocatalytic process.

INFLUENCE OF DIFFERENT ANNEALING TEMPERATURES AND ATMOSPHERE ON THE PROPERTIES OF Bi_2O_3 NANOPARTICLES

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The metal oxides of the A_2O_3family are materials used in a wide variety of applications due to their exceptional properties, such as high chemical, mechanical and thermal stability. Among these oxides, bismuth oxide (Bi_2O_3) has attracted the attention of several research groups due to its high technological potential for applications in photocatalytic processes, besides being a material-rich polymorphism that allows several phase-dependent physicochemical properties and technical applications [1-3]. However, the synthesis of Bi_2O_3polymorphs requires expensive and difficult-to-reproduce routes [2]. Thus, in this work, we studied the synthesis of Bi_2O_3nanoparticles using the solvothermal method, in which we sought to evaluate the influence of different annealing conditions on the properties of Bi_2O_3. The precursors used in the synthesis were Bismuth nitrate pentahydrate, ethyl alcohol, and sodium hydroxide. The samples were annealed at room pressure and at 10 mtorr, using the following temperatures: 300°C, 400°C, 500°C, 600°C, 700°C and 800°C. Preliminary characterizations indicate that some samples present a mixture between two phases: the cubic phase of $Bi_2O_3$and the monoclinic phase of Bi_2O_3, which are directly influenced by the calcination temperature.

Acknowledgments: We thank the FAPESP for granting the scientific initiation scholarship (2022/01901-0; 2022/02994-20) and CNPq (310778/2021-2). To the Instituto Tecnológico de Aeronáutica (ITA) and the Instituto Federal de São Paulo (IFSP) for providing the infrastructure used during the project execution. And to the whole team of the Laboratory of Plasmas and Processes (LPP - ITA) for the support.

Influence of the CN-HAp percentage on hidroxyapatite composite synthesized from eggshells for photodegradation of dyes

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Heterogeneous photocatalysis is a method capable of degrading chemical species, such as dyes, from industrial processes, from the absorption of photons by a semiconductor, where obtaining an efficient photocatalyst can be done from the synthesis of heterostructures, which combine energetically. g-C3N4 is a semiconductor that can be easily synthed from the calcination of urea and other low-cost products. Despite having acceptable bandgap, its rapid recombination of photoinduced electron-hole pairs and low surface area make its application difficult for photocatalysis. On the other hand, hydroxyapatite is a calcium phosphate, which can be extracted from biogenic sources, such as eggshells, but which has a high bandgap value. Thus, the synthesis of heterostructures combining CN-HAp was proposed using the hydrothermal method with microwave irradiation. Hydroxyapatite and g-C3N4 were produced from eggshell and urea calcination, respectively. The photocatalytic performance under sunlight and UV irradiation was evaluated for different dyes, as well as the reuse of photocatalysts, simulating real conditions, which implies a high potential for practical application.

Acknowledgment

PIBIC e LSQM - UFRN

References

[1] T. Xu, R. Zou, X. Lei, X. Qi, Q. Wu, W. Yao, Q. Xu, New and stable g-C3N4/HAp composites as highly efficient photocatalysts for tetracycline fast degradation, Appl. Catal. B Environ. 245(2019)662-671.

[2] Y. Jim´enez-Flores a,b , K. Jim´enez-Rangel a , J.E. Samaniego-Benítez a,c , L. Lartundo-Rojas d , H. A. Calderon´ e , R. Gomez ´ b , A. Mantilla. Novelty g-C3N4/HAp composite ashighly effective photocatalyst for Cr (VI) photoreduction. Catalysis Today388-389(2022)168-175.

Insights into the modified HAp-based photocatalytic process and its challenges

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This review describes the applications of HAp compounds and derivatives in the degradation of organic pollutants, with emphasis on the removal of dyes, drugs and pesticides in modification studies and their properties and stability.The fundamentals of heterogeneous photocatalysis were accepted, combined with operational parameters that worked on HAp degradation, strategies and modification functions (doping, catalytic support, heterojunction). Furthermore, emphasizes, discusses and reports the state of the art of these promising compounds for use and insights into their efficiency, ease of synthesis and nature to remove pollutants from wastewater streams, primarily using heterogeneous photocatalysis as an active methodology in wastewater destruction. These compounds are considered to be efficient photocatalysts due to their ease of removing contaminants from wastewater using advantageous redox activity and other flexible properties. The synergistic effect is explained due to structural changes that minimize ecological and environmental problems. According to the knowledge acquired so far, in addition to the numerous applications of HAp in tissue engineering, doped, supported, constructed and heterostructured HAp were found in photodegradation studies. Therefore, considering the rapid advances in technology in the discovery of new compounds, many promising indicators can be applied to raise even more challenges in science and research
.Acknowledgements: The authors would like to thank CAPES, CNPq, FAPEPI.

Investigation of photochemical and photophysical properties of S/Se-Terchalcogenophene-C_{60} dyads

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The increasing popularity of fullerenes and their derivatives over the past few years is due to the fact that they exhibit interesting properties that include high affinity of electrons and the ability to be excited with high-energy light [1]. Fullerenes have good conductive properties, large specific surface area, high chemical and thermal stability. These features make them attractive for application in variety of areas, like photovoltaic devices, as catalysts in the synthesis of fine chemicals or photodynamic therapy [2].

This work presents a study devoted to fullerene C_{60} derivatives as promising photosensitizers. In order to determine their usefulness, spectroscopic measurements were carried out and their quantum yield of singlet oxygen photogeneration was estimated. Moreover, the results made it possible to evaluate the effect of functionalization of fullerene core on its photosensitizing properties. It has been shown that dyads due to the presence of these thiophene-based lancets are more effective than unsubstituted C_{60}in white light. Selected C_{60}dyads were used as a source of singlet oxygen in a model DHN Photooxidation reaction in a batch photoreactor.

Acknowledgements:

This work was funded by National Center of Science, grant no. 2016/21/D/ST5/01641. Authors acknowledge the EU’ s Horizon 2020 for funding the OCTA project under grant agreement No. 778158. The work was supported by the Polish Ministry of Education and Science funds for science in 2018-2023 allocated to the implementation of an international cofinanced project.

References:

[1] Dirk M. Guldi, Accounts of Chemical Research, 2000, 33(10), 695-703.

[2] Yuan Pan, Applied Catalysis B: Environmental, 2020, 265

Investigation of Supercapacitor Properties using Iron Tungsten Porous Electrode in aqueous electrolyte

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Porous supercapacitors (SCs) materials are great interesting for energy storage system [1]. This paper, iron tungstate mixed oxides electrode was prepared by polymeric precursor method and deposited onto conductor substrate by drop casting heat-treated at 500 ºC for 2 h. The thermal, structural, and morphological characterization was realized by differential scanning calorimetry (DSC), X-ray diffraction (XRD), emission-scanning electron microscopy (FE-SEM), atomic force microscopy (AFM), respectively. Crystallization process of polymeric resin occurred at 454°C. Synergistic effect of monoclinic FeWO4 and monoclinic Fe2W3O12 phases were confirmed with Fe/W mixed oxides. An irregular shape of condensed spherical-like particles aggregated with rough and porous nanostructures were observed. SC electrochemical analyses of Fe-W electrode accomplishes a capacitance of 75 F g-1 at 5 mV s-1, energy density of 3.73 Wh kg-1, power density of 582.1 W kg-1and long-term electrochemical cycling with a capacity retention of 94.04% after 6000 cycles. Therefore, Fe-W based electrode demonstrates a potential supercapacitor device with excellent stability in a nontoxic aqueous electrolyte.

References

[1] N. M. Shinde, P. V. Shinde, R. S. Mane, K. H. Kim, Solution-method processed Bi-type nanoelectrode materials for supercapacitor applications: A review. Renewable and Sustainable Energy Reviews 135 (2021) 110084.https://doi.org/10.1016/j.rser.2020.110084

Lanthanum ferrites synthesized by solution combustion synthesis (SCS) using sucrose as fuel.

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Abstract Lanthanum ferrites present electrical and magnetic properties that enable their use in several areas. Specifically, strontium and cobalt doped lanthanum ferrites have electrical conductivity and thermal stability, which enable their use, for example, as electrodes in solid oxide fuel cells [1-3]. This study aimed to synthesize $La0,6Sr0,4Co0,8Fe0,2O3$ nanoparticles powders through solution combustion synthesis using sucrose as fuel aiming several applications. The excess of cobalt in the composition provides greater conductivity. The $La0,6Sr0,4Co0,8Fe0,2O3$ powder was obtained using oxidant and reductant reagents in a stoichiometric amount and in excess of reductant reagent in the composition. In sequence, thermal treatment in several temperature ranges was performed. As a result, the samples calcined at 1200°C showed the best structural characteristics in relation to the other temperatures used, but with the largest crystallite size. Also evaluated were the electrical properties. The conductivity of the materials tends to increase with increasing temperature and excess of fuel.

Acknowledgments

The authors acknowledge the Coordination for the Improvement of Higher Education Personnel), Brasil ((CAPES) - Financial Code 001) for their financial support.

References

[1] M. Lubini, E. Chinarro, B. Moreno, V. C. de Sousa, A. K. Alves, C. P. Bergmann, Electrical Properties of $La0.6Sr0.4Co1-yFeyO3$ (y = 0.2-1.0) Fibers Obtained by Electrospinning. The Journal of Physical Chemistry C. V.120(1) (2016), 64-69.

[2] S.P. Jiang, Development of lanthanum strontium cobalt ferrite perovskite eletrodes of solid oxide fuel cells - A review. Int. j. of hydrogen energy, V. 44 (2019), 7448-7493.

[3] A. Mazrouei, A. Saidi,Microstructure and magnetic properties of cobalt ferrite nano powder prepared by solution combustion synthesis. Materials Chemistry and Physics, V. 209 (2018), 152-158.

Macroporous monoliths of silica/niobium pentoxide obtained by spinodal decomposition during sol-gel transition: adsorption dependance on microstructure.

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Macroporous silica/niobium pentoxide composite monoliths were synthesized by sol-gel method. Macroporosity was induced by spinodal decomposition, using PEG 10000 as soluble polymer, triggered by the presence of ammonium niobate(V) oxalate in the precursor solution as described elsewhere [1]. Monoliths were calcinated at 500, 700 and 900 °C Composite structure was characterized by XRD, MEV, HRTEM, N2 adsorption. Macroporous interconnectivity was studied by methylene blue adsorption, after adsorbing the dye for three, six and nine days, the monoliths were cut in half for evaluating the internal surface of the material, the progression of the color towards the center of monoliths showed the interconnectivity of pores. By MEV the macropores are in the range of 1 to 3 μm, by HRTEM crystallite size of niobium pentoxide showed dependance on calcinating temperature, ranging from 4 nm at 500 °C, to 8 ± 2 nm at 700 °C, to 16 ± 6 nm at 900 °C. The d spacingof planes at HRTEM were in accordance with XRD data and at 700 and 900 °C the predominant phase of niobium pentoxide was the orthorhombic. Surface area also showed dependance on calcinating temperature, ranging from 705 m²/g at 500, to 502 m²/g at 700, to 211 at 900 °C. The composite was studied for Doxycycline adsorption at pH range from 2to 12, in all the cases, sample calcinated at 700 °C showed better performance than samplescalcinated at 900 °C and calcinating at 500 °C lead to the lowest adsorption. Maximum ofadsorption at pH 9, in sample calcinated at 700 °C (50 mg/g). This result indicates that forthis type of composite not only the highest surface area maters for great adsorption, structural properties (as crystallite size and zeta potential) are also important.

Acknowledgments: CNPq, FINATECH, FAPEMIG, CAPES, Center of Microscopy/UFMG.

References:[1] Lima, L. F. S., Mohallem, N. D. S. J. Porous Mater 28, 361-367 (2021).

Membrane scaling in electrodialysis for fluorideremoval from groundwater

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This research deals with the natural contamination of groundwater by fluoride(F-) and the application of the electrodialysis(ED) technique to remove F-. Fluoride contamination is a recurring issue all over the world. International health organizations establish 1.5 mgF-/L as the limit in drinkable water. Conventional water treatment is ineffective in removing F-, which makes it necessary to use alternative techniques, such as ED. This process apply ion-selective membranes and electric potential as the driving force to remove F-. During the ED tests, parameters were monitored, including conductivity, F- concentration, potential and current, to evaluate the performance of the process. A synthetic solution simulating natural groundwater containing 5.5 mg/L of F- and other ions that compete with F- transport was treated by ED. The results showed a demineralization of 86.9% with a 0.005σ. However, there was an unexpected behavior regarding the concentration of F- It remained stable in the range of 10 to 11 mg/L after the 4° cycle of concentration. This behavior can be explained by a phenomenon scaling, which is the precipitation of inorganic salts on the membrane surface, and could be intensified by concentration polarization [1]. To confirm this assumption, scanning electronic microscopy(SEM) images for new and post-use anionic membranes were obtained. Magnified 1000x, the SEM images evidenced precipitate on the membrane. The analysis of energy dispersion X-ray spectroscopy obtained coupled to SEM indicated the precipitate composition, including F and Ca for the post-use membrane. A chemical speciation diagram obtained using the Hydra Medusa software had also confirmed the formation of CaF_2. Furthermore the solubility of CaF_2 is 16mg/L[2]. The study indicatedthat ED is an effective technique to remove F-, but there are still challenges to be beat forits large-scale. An alternative is to evaluate the ED reversal, aiming to reduce polarization concentration and scaling.

Microwave-assisted synthesis of Carbon Dots from glycerin residue and its photophysical studies

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Carbon Dots (CDs) are nanomaterials smaller than 10 nm with unique electrical and optical properties like tunable photoluminescence, light absorption over a wide range, and higher electrical conductivity. CDs are versatile and can be applied in biomedicine, optoelectronics, sensing, catalysis, etc. Several organic precursors can be used to prepare the CDs, including glycerin. However, glycerin as a by-product in biodiesel production has still not been explored in synthesizing CDs. Glycerin residue can impact the economic and environmental viability of biodiesel, and research for the application of this by-product, particularly in a green and sustainable way, are necessary. In this study, CDs from crude glycerin were prepared by microwave-assisted synthesis and applied as photocatalysts CDs from pure glycerin were also synthesized for comparison. Hexamethylenediamine was used as a dopant in both cases to increase the emission. The synthesis was carried out in a microwave oven (PMM24, Philco) using a power of 504 W and irradiation time of 3 minutes. For the photophysical study, absorbance, fluorescence emission, and excitation measurements were conducted in a spectrofluorometer (FS-5, Edinburgh Instruments). Results showed more considerable emission (at λ_{exc}=380 nm) by CDs from the crude glycerin doped with hexamethylenediamine. These CDs showed one broad emission band with three peaks at 415 nm, 438 nm, and 462 nm. The CDs were impregnated with TiO_2, and the photocatalysis activity was observed in the paracetamol degradation. Thus, we believe that crude glycerin can be used for the production of functional photocatalysts to be used in the remediation of contaminants.

Acknowledgements: FAPESP (Processo 2019/13471-8).

Mineral residues of the state of Paraíba: evaluation and application as photocatalytic support

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The Borborema Province in the Northeast of Brazil is characterized by the presence of mineral resources such as kaolin. Its processing generates residues rich in kaolinite, mica and quartz, which need to be managed and reused. Therefore, this study aims to add value to these residues by its use in photocatalysis as support materials for TiO_2, a photocatalytic material. The residues were characterized by x-ray diffraction (XRD) and energy dispersive spectroscopy (EDS), and subsequently they were submitted to mechanical agitation and particle size separation. After this process, the $TiO_2$was deposited in the residual quartz by the modified Pechini Method. Titanium citrate was used as a precursor for the synthesis of the polymer resin when reacting with ethylene glycol. The resin underwent a first calcination at 300°C, and subsequently a second calcination was performed at four distinct temperatures (500°C, 600°C, 700°C and 800°C)[1]. The calcined materials were characterized by XRD, ultraviolet-visible spectroscopy and infrared spectroscopy. The formation of anatase and rutile in different proportions depended on temperature, with band gap value varying between 2.6 and 2.9 eV. To attest the photocatalytic efficiency, eachmaterial was tested in a UVC reactor in reaction with terephthalic acid (TA), a probe forhydroxyl radicals. The aliquots were analyzed in a spectrofluorimeter to evaluate the formation of 2-hydroxyterephthalic acid, a fluorescent compound, originated due to the reaction of ●OH with TA [2]. The results of photocatalytic tests attested to a greater formation of ●OH for the synthesized material at a temperature of 600°C, while quartz isinert to photocatalysis.

Acknowledgments:
CAPES/MEC, FINEP/MCTI,PIBIC/CNPq/MCTI,PRONEX/FAPESQ-PB/CNPq/MCTI

References:
[1] L. DIMESSO. Handbook of Sol-Gel Science and Technology, v.1, p. 1-22, 2016.
[2] L. RAO et al, Materials Research Bulletin, v. 57, p. 41-46, 2014.

MoO3 applied in the discoloration of textile dyeaqueous solution

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The project involves the synthesis of molybdenum trioxide by the microwave-assisted hydrothermal method, with orthorhombic or hexagonal structure, and application in the photocatalytic discoloration of methylene dye in aqueous solution. In the last years MoO3 has been studied for the photodegradation of several dyes, including MB, with good results. In the present work, photocatalytic and adsorptive tests were carried out, and the interaction between molybdenum trioxide and methylene blue was evaluated as well the photohydroxilation of terephthalic acid (TA), used as probe for hydroxyl radicals, to verify if the photocatalytic process really occurs. The tests with MB were performed using the dye in solution at three different values of pH, 4, 5.6 and 8, as the solution pH determines the amount of the protonated dye molecules and consequently the interaction between dye and substrate. Very good results were obtained at pH 4 with values above 90% for discoloration for adsorption or photocatalysis after 8 h of test, for both MoO3 phases, whereas the orthorhombic phase showed a faster reaction kinetics compared to the h-MoO3. Nonetheless, the h-MoO3 showed good values of discoloration for photocatalysis in pH 8 after 8 h, with a growth of approximately 5-fold comparing to adsorption. For all samples photodegradation takes place, but the discoloration occurs by a direct photocatalytic process, in other words, the photocatalytic process occurs due to direct electron transfer between dye and MoO3 without the formation of hydroxyl radicals, as indicated by the small amount of hydroxyl radicals in solution in the presence of MoO3 indicated by the photohydroxilation of TA under UVC irradiation.

Acknowledgments: PRONEX/CNPq-MCTIC/FAPESQ, FINEP/MCTI, PIBIC/CNPq/UFPB

References:

[1] Chithambararaj, A; Chandra, A Rajeswari, A Y. Am. Chem. Soc., 16: 1984-1995 (2016)

Nano-ZnO synthesized by the sol-gel route withdifferent starches

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The aim of this work was to investigate the influence that starches from different botanical sources have on some properties of zinc oxide synthesized by the starch-assisted sol-gel route [1]. In this sense, three ZnO powders were synthesized so that cassava, potato and corn starches were used in the synthesis as stabilizing agents for the formation of nanoparticles. In general, it was observed that the type of starch has a certain influence on the crystallite size, as well as on the band gap value. The smallest crystallite size (20 nm), calculated by the Scherrer formula, was found in ZnO whose starch used in the synthesis was cassava, as well as the largest band gap (3.16 eV), calculated by the tauc method, was obtained for this same sample. Based on literature values, cassava, potato and corn starches have amylose contents of 17%, 21% and 26% respectively [2]. From this, it was noticed that when using starch with a lower amylose content, we obtained a ZnO with a smaller crystallite size and a higher band gap value.

References:
[1] Almeida WL, Rodembusch FS, Ferreira NS, Sousa VC (2020) Eco-friendly and cost-effective synthesis of ZnO nanopowders by Tapioca-assisted sol-gel route. Ceram Int. https://doi.org/10.1016/j.ceramint.2020.01.095
[2] Stephen AM, Phillips GO (2006) Food Polysaccharides and their Applications, 2nd edn. CRC Press, Boca Raton. https://doi.org/10.1201/9781420015164

$Ni_{0.3}Cu_{0.2}Zn_{0.5}Fe_2O_4$ Ferrite as Photocatalysts using Solar Energy

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$Ni_{0.3}Cu_{0.2}Zn_{0.5}Fe_2O_4$ ferrite was evaluated as photocatalyst in the degradation performance of industrial effluent methylene blue (MB) using solar photocatalysis. Ceramic materials can be a promising field of research extremely attractive in renewable energy and with great industrial possibilities. The photocatalyst was produced by combustion reaction, a method chosen for presenting a low energy consumption, being fast to manufacture [1]. The experiment was done using Ni-Zn ferrite doped with 2% Cu ions ($Ni_{0.3}Cu_{0.2}Zn_{0.5}Fe_2O_4$) [2]. The best conditions of the variables during the solar heterogeneous photocatalysis process were 10 mg/L of MB, 2.0 g/L of photocatalyst, 408 mg/L of H_2O_2 and pH 3. The photocatalyst studied combinated to such parameters reached an efficiency of 79.3%, in terms of color removal and dye concentration, demonstrating to be an efficient photocatalysts for degradation of MB. A deeper statistical planning, in order to determine the best conditions of the variables studied and to find the optimal point for degradation of the MB dye will be developed.

Acknowledgements:
CNPq, Propesq-UFPB

References:
[1] S.M. Aydoghmish; S.A. Hassanzadeh-Tabriz; A. Saffar-Teluri, Ceramics International v. 45(12), p. 14934-14942, (2019).
[2] J. Dantas, E. Leal, A.B. Mapossa, D.R. Cornejo, A.C.F.M. Costa. Fuel (Guildford), v. 191, p. 463-471, (2017).

Obtaining dense $Ba_5Nb_4O_{15}$ ceramics at lower sintering temperatures

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$Ba_5Nb_4O_{15}$(BNO) ceramics have been extensively investigated for their dielectric and luminescence properties. Furthermore, this material displays protonic conduction and has potential to be used as electrolyte in solid oxide fuel cells (SOFCs) [1]. SOFCs are important devices in changing the energy matrix. However, producing dense BNO ceramics for use as electrolytes in SOFCs presents difficulties, since high sintering temperatures can result to elongated grain growth and even de-sintering phenomena [2]. Therefore, in this work, it was employed the solid-state reaction synthesis with successive milling and calcination steps. This synthesis process already allowed an increase in densities at lower sintering temperatures. The sintering conditions used included the conventional method using sintering aids, as well two-step sintering of pristine BNO. The X-ray diffraction patterns of calcined powders revealed a single phase of $Ba_5Nb_4O_{15}$. The samples sintered using sintering aid, 1-3 wt.% ZnO, achieved satisfactory densification of 95% at a sintering temperature of 1200 ºC, but SEM images revealed abnormal grain growth. On the other hand, the samples with NiO resulted in sample with elevated porosity. For two-step sintering, the first step temperature was 1250ºC, and both second step conditions, at 1200 and 1150ºC, achieved densities of 92%. However, when the second step is 1150ºC, a more refined microstructureis obtained.

Acknowledgements:

CAPES, Fundação Araucária, CNPq and C-labmu.

References:

[1] L. F. Luiz, E. C. Grzebielucka, A. S. Antonio Chinelatto, G. C. Mather, and A. L. Chinelatto, "Synthesis and electrical characterization of $Ba_5Nb_4O_{15}$and $Ba_5Nb_{3.9}M_{0.1}O_{(15-\delta)}$ (M = Ti, Zr) hexagonal perovskites," Ceram. Int., vol. 45, no. 4, pp. 5087-5092, 2019.

[2] C. Y. Tsao, W. H. Tuan, and K. C. Feng, "De-sintering of $Ba_5Nb_4O_{15}$ceramic and its influence on microwave characteristics," J. Eur. Ceram. Soc., vol. 37, no. 4, pp. 1517-1521, 2017.

Obtainment and Characterization of Ceramic Electrospun Nanofibers for Antimicrobial Applications

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The technological advances have increasingly and the preparation of materials with unique properties is required to meet functional needs. In this work, powders of $Ag_2WO_4$and Ag_2MoO_4, which were previously synthesized by the co-precipitation method, were incorporated at 50 wt-% into the matrix of poly(vinyl alcohol) (PVA) (conc. 10 wt-%) for preparation of composite ceramic nanofibers, using the electrospinning method. The obtained materials were structurally characterized by DRX, and the morphology was evaluated by SEM, for further studies of photocatalytic and antimicrobial properties. DRX profiles of $Ag_2WO_4$and Ag_2MoO_4powders indicate the prevalence of β-$Ag_2MoO_4$and α-Ag_2WO_4phases, respectively, and that the peaks remain very sharp in DRX of PVA/$Ag_2MoO_4$and PVA/Ag_2WO_4composite nanofibers. SEM images show that powders and electrospun nanofibers possess non-uniform distribution. Electrospinning was effective in the preparation of PVA/$Ag_2MoO_4$and PVA/Ag_2WO_4fibers. Antimicrobial and photocatalytic activities essays are ongoing. Antimicrobial activities of powders and polymeric mants against Pseudomonas aeruginosa and Staphylococcus aureus were investigated through the minimum inhibitory concentration (MIC) and the minimum bactericidal concentration (MBC) [2]. The results indicate that the materials have strong antimicrobial potential of action.

Acknowledgements: UFPI, LIMAV, IFMA, CNPq, CAPES and FAPEPI.

References

1 - Assis, M.; Robeldo, T.; Foggi, C. C.; Kubo, A. M.; Mínguez-Vega, G.; Condoncillo, E.; Beltran-Mir, H.; Torres-Mendieta, R.; Andrés, J.; Oliva, M.; Vergani, C. E.; Barbugli, P. A.;Camargo, E. R.; Borra, R. C.; Longo, E. Ag Nanoparticles/α-Ag_2WO_4Composite Formed by Electron Beam and Femtosecond Irradiation as Potent Antifungal and Antitumor Agents. Sci. Rep., v. 9, p. 9927, 2019.

2 - Balouiri, M, Sadiki, M.; Ibnsouda, S.K.; Methods for in vitro evaluating antimicrobial activity: A review. Journal of Pharmaceutical Analysis. V. 6, p. 71-79, 2016.

One-pot electrosynthesis of hierarchical TiO2/rGO nanocomposites for green hydrogen production

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The conversion of solar energy into chemical energy via photoelectrocatalysis is considereda promising alternative in the face of the current energy crisis.[1] We report the synthesisvia electrochemical anodization of TiO2 nanotubes with graphene oxide and in situ reduction by thermal route in an oxidizing and inert atmosphere (N2 controlled flow).[2] We investigated the effect of thermal reduction of graphene oxide (GO) on the photoelectrocatalytic properties of the TiO2/rGO composite in order to improve light absorption. The photoanodes produced were characterized by Raman Spectroscopy and SEM, in addition to their photoelectrochemical evaluation. The TiO2 photoanodes showed a photocurrent density of 0.075 mA.cm-2 and resistance of 5.93 kΩ in anatase phase. TiO2/rGO treated in an ambient and inert atmosphere has a photocurrent density of 0.135 and 0.480 mA.cm-2, 1.8 and 5.4 times greater than pure TiO2, respectively. A system resistance reduction was also observed for both systems, going to 3.7 kΩ after heat treatment in an inert atmosphere. The heat treatment atmosphere interferes with the quality of the photoanodes produced, due to the difference in the degree of reduction of the GO structures anchored in the nanotubes. This behavior is evidenced in Raman spectroscopy, where a reduction in the degree of structural disorder is shown after thermal treatment in an inert atmosphere, reinforcing the hypothesis of GO conversion into rGO.[3] These results suggest that treatment in an inert atmosphere promotes a more efficient reduction of GO directly in the TiO2 nanotubular matrix, restoring the typical structure of graphene and, consequently, improving the photoelectrochemical performance.

[1] Zheng, et al., Appl. Surf. Sci., 512, 144549 (2019).
[2] Soares et al., J. Electroanal. Chem., 902,115802 (2021).
[3] Wang, et al., Symmetry., 12, 1420 (2020).

One-step nickel-titanium dioxide nanotube matrix synthesis by pulsed anodization

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Water quality is a pressing issue for present and future generations, highlighted by the UN''s 2030 Agenda for Sustainable Development [1]. In Brazil''s northeast, textile industries are a major source of water contamination due to recalcitrant dyes, exacerbating the limited water supply. Improved technologies are necessary for enhanced efficiency of water treatment systems. Advanced oxidation processes (AOPs) like photocatalysis hold promise for effective treatment, but commonly used semiconductors like TiO_2have limited responseto visible and sunlight, necessitating modifications for effective operation. To obtain a nanostructured material based on Ni@TiO_2nanotubes, this study optimized pulsed anodization [2-3] parameters (potential and deposition time) using the design of experiments (DoE). A two-electrode system was used, with a titanium foil and copper plate as anode and cathode respectively, connected to a sourcemeter model 2400 (Keithley) and controlled by Labview (National Instruments). The electrodes were fixed at 1 cm and immersed in an electrolyte solution (0.7% ammonium fluoride (NH4F) with 100 mM of Ni^{2+}dissolved in 90:10 v/v mono ethylene glycol:water) under stirring (200 rpm). Anodization parameters were fixed at 30 V for 5 s followed by deposition conditions. Photocurrent density (mA/cm2) and methylene blue photodegradation (%) were evaluated as responses during the DoE and reported significant effects of both deposition parameters. **Acknowledgements**: This work was supported by FNDCT through Finep [process 0122056900] and by Facepe [APQ-1237-1.06/21]. PCS is grateful to Facepe for her scholarship [BIC-0248-3.07/22]. VNL, CEML and BRSI are grateful to CNPq for their PCI fellowship and PhD scholarship [process 140123/2022-9]. **References**: [1] UN, The 2030 Agenda for Sustainable Development. [2] T. Soares, T. A., et al. Materials Letters, 284, 128975. (2021) [3] R.A. Galvão et al. Journal of Materials Research, 34(11), 1868-1878. (2019)

Optimization of Heterogeneous Solar Photocatalysis using the Design of Experiments with Ni0.2Cu0.3Zn0.5Fe2O4 Nanoferrite in the Demineralization Process of Methylene Blue Textile Effluent

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Recently, new photocatalytic materials, such as doped and heterostructured materials, have been studied, as these systems have great potential for the development of heterogeneous photocatalysis powered by solar energy for environmental remediation [1]. Heterogeneous solar photocatalysis has received increasing attention for presenting important environmental applications, being an efficient technology in the degradation of pollutants in water and synthetic dyes from textile dyeing. The objective of the study was to use modeling and optimization to statistically analyze the demineralization of textile effluent with methylene blue dye (MB), using NiZnCuFe nanoferrite as a semiconductor photocatalyst (dopping of 30% Cu) and solar energy as an energy source. For this, a full factorial experimental arrangement with three input variables (2k = 23) and insertion of center points based on Response Surface Methodology (RSM) was used. The response variables fitted well to the evaluated tests, obtaining significance in the parameters related to pH and hydrogen peroxide concentration. It was found that the optimal values for the photocatalyst would be 0.1 g/L; hydrogen peroxide 120 mg/L and pH at 5, in which under these conditions a maximum yield of 73.83% of degradation of the MB dye was reached.

Permeable concrete with ZnO nanoparticles applied tothe treatment of industrial effluents

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Emerging contaminants (ECs) from inappropriate industrial disposal, are a constant concern today, due to its tendency to degrade the aquatic environment and cause a serious threat to living organisms. Nowadays, dyes are present as one of the main alarming sources of water contamination from the industry. Studies on the photodegradation of contaminants in wastewater using nanomaterials have stood out as an environmentally correct process that completely destroys pollutants [1,2]. Combining this property in the industrial structure has been a challenge for engineers and scientists. Permeable concrete (PC) is a material composed of an abundant volume of interconnected pores, allowing water to pass freely through the brick. As result, new technologies are being applied to this concrete to improve the quality of the water that passes through and reaches the ground, as well as treatment that allows the PC to act in the decontamination of water [3]. In this work, permeable concrete bricks with controlled porosity will be produced and subsequently impregnated with zinc oxide nanoparticles (ZnONPs) with photocatalytic properties synthed by hydrothermal method for the treatment of effluents. The concentration of dye; dosage of nanoparticle; pH effect, and contact time were evaluated using only ZnONPs. The methylene blue dye (MB) photodegradation under UV-Vis light reached 73% at pH 6 and 92% at pH 10, with 10 mg of nanoparticle and a dye concentration of 10 mg/L.

Acknowledgments

The authors would like to thank the support of UNESP, the Brazilian agencies FAPESP (Sao Paulo Research Foundation), and CAPES (Coordenação de Aperfeiçoamento de Pessoal deNível Superior).

1. Punia P, et al. (2021) Ceramic Int. 47:1526-1550
2. Chong MN, et al. (2010) Water Res. 44:2997-3027
3. Teymouri E, et al. (2020) Jounal Water Proc. Eng. 36:101346

Permeable Reactive Barrier for soil remediation using Sodium Polyacrylate

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The growing contamination of ecosystems from industrial activities is a problem to be solved by organizations and society. Despite state and federal laws, the incorrect disposal of contaminated waste in soils and watercourses continues to be a habit and, over the years, the consequence is the formation of environmental liabilities, especially in industrial areas [1]. The present work aimed to establish a method for remediation of soils contaminated with toxic metals, using a permeable reactive barrier (PRB) [2]. To demonstrate the effectiveness of sodium polyacrylate (PAAS) in the adsorption of the contaminating metals, a leaching test was carried out. For this, the samples were mounted vertically in 1.5L PET bottles with layers of contaminated soil, polymer and standard soil. The layer of ''swollen'' or original powdered sodium polyacrylate was placed between the two soils to act as a barrier. The system was kept for 30 days and disassembled for further analysis using Inductively Coupled Plasma (ICP) to detect metallic elements. The results showed a reduction in the levels of Ba (42.87%), Pb (70.37%) and Fe (48.63%) in the contaminated soil, by transferring it to the PAAS, where the metals were retained. The highest transfer and adsorption occurred in the assembled system with the polymer in powder form. In this way, the PAAS proved to be efficient in the removal of contaminating metals from the soil, such as a PRB, at least for those analyzed. The resulting polymer residue could be used in the cement industry, in line with circular economy principles.

Acknowledgements:
To FAPESP (Process No 2022/13496-3) and to Faculdade de Tecnologia de Sorocaba.

References:
[1] RIBEIRO, L. G. G. and Iasbik, T. A. *Argumentum*v. 20, page 165-184 (2019)
[2] CORÁ, A. M. *et al.*Boletim Técnico da FATEC-SP v. 54, page 54 (2022)

Photocatalytic activity of Nb-doped TiO_2 thin films deposited by spin coating

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Since the discovery of the high electrical conductivity of Nb-doped TiO_2films [1], investigations of this material have been addressed toward applications as transparent conducting oxides [2] and thermoelectricity [3]. In this work, the catalytic activity of Nb-doped TiO_2thin films from degradation of methylene blue (MB) is investigated. Films composed by $TiO_2$and Nb_2O_3nanoparticles were deposited on polished aluminum substrates by spin coating as function of the Nb_2O_3concentration (0, 2.5 and 5.0% wt.). Experiments of the catalytic activity were carried out in a homemade experiment composed by a ramp where the MB particles, dissolved in deionized water, flow over the substrate surface with controlled speed under UV irradiation. The optical properties of the tested solutions, such as absorbance and degradation efficiency, were evaluated by UV-Vis spectroscopy as a function of the degradation time. Samples were also analyzed by X-ray diffraction, field emission scanning electron microscopy, X-ray photoelectron spectroscopy and four-point probe to investigate the crystalline structure, morphology, chemical composition and electrical properties, respectively. Films are polycrystalline with preferential anatase phase, 30 μm average particle size with homogenous distribution, no structural failures, low Nbconcentration and influence of the Nb content on the MB degradation.

Acknowledgments:

D. A. Duarte thanks CNPq for financial support (grant nº 307408/2021-3).

References:

[1] Y. Furubayashi et al., Appl. Phys. Lett., 86, 252101 (2005).

[2] J. M. Ribeiro et al., J. Alloys Compd., 838, 155561 (2020).

[3] J. M. Ribeiro et al., Surf. Coat. Technol., 425, 127724 (2021).

Photocatalytic activity of $SrSnO_3$/Ag composites – use of scavengers to evaluate the reactive species during the photohydroxilation of terephthalic acid

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The mitigation of pollutants in general has become one of the most challenging and critical tasks for the entire scientific community [1]. In this context, this work aims to obtain materials with $SrSnO_3$structures superficially modified with $Ag^+$and formic acid for the evaluation of photocatalytic performance by the photohydroxylation of terephthalic acid. The $SrSnO_3$was obtained by the method of modified-Pechini method, with calcination at 900°C, while the solid $SrSnO_3$/Ag was obtained by photodeposition of Ag under UVC radiation and drying for 24 hours at 80°C. The obtained powders were characterized by X-ray diffraction (XRD), infrared spectroscopy (IR), ultraviolet-visible spectroscopy (UV-vis), field-emission scanning electron microscopy (FE-SEM) coupled to energy dispersion X-ray spectrometry (EDS). The photocatalytic applications suggest a satisfactory performance in the photohydroxylation of terephthalic acid by both systems mentioned above, with emphasis on the $SrSnO_3$/Ag system. The use of scavengers such as isopropanol, EDTA, and N_2revealed in more detail the oxidative species responsible for the photocatalytic performance both for the $SrSnO_3$system and for the $SrSnO_3$@Ag composite, as well as the possible photocatalytic pathways that followed the photohydroxylation reactions and generation of the fluorescent compound 2-hydroxy-terephthalic acid, which was monitored by spectrofluorimetry.

Acknowledgments: FINEP, CAPES, PRONEX/FAPESQ/CNPq

References:[1] LUO, J. and ZHANG, W. EcoMat, v. 3, p. e12079, 2021.

Photocatalytic degradation of dyes using the MIL88A/MoS2 heterojunction

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Organic pollutants, such as dyes, have been discharged into the environment by the industrial sector, with negative impacts on water resources and ultimately, posing risks to the human health.(1) Photocatalysis presents a viable solution to this problem by effectively converting solar energy into chemical energy, thereby facilitating reactions that contribute in the removal of these contaminants from water resources.(2) Metal-organic frameworks (MOFs) are 3D porous structures that have been recently explored in photocatalytic applications, as well as modified MOFs, to enhance the effect of photocatalysis.(3) Here we synthesized MOF MIL88A and MIL88A/Molybdenum disulfide (MoS2) composite to compare their efficiency and investigate the formed heterojunction in photocatalytic activity. Subsequently, to facilitate the recovery of the catalyst, the particulate was immobilized on C/SiO2 ceramic fibers obtained via the solution blow spinning (SBS) method. The morphological aspects of MOF/MoS2 supported on C/SiO2 were determined by scanning electron microscopy (SEM) techniques. The crystalline and chemical structures were determined by X-ray diffraction (XRD) and infrared spectroscopy (FTIR), respectively. The materials' photocatalytic activity was investigated towards methylene blue (MB) and rhodamine B (RhB) solutions as model dyes using UV-Vis spectroscopy. The catalytic activity of the MOF was tested in solutions of MB and RhB, achieving efficiencies of 48.5% and 58.92%, respectively, while the MOF/MoS2 exhibited superior performance with efficiencies of 90.04% and 71.73%. The ceramic fibers C/SiO/MOF/MoS2 yielded results of 37.25% for MB and 24.59% for RhB. Acknowledgments: the authors thank to CAPES, CNPq, FAPESP and Embrapa. References: (1) Jeong C. et al. Separation and Purification Technology, vol 305, (2023). (2) Song, C. G. et al. ACS Appl Nano Mater, vol 5, 18930-18939 (2022). (3) Li, S. et al. Journal of Environmental Chemical Engineering, vol 9, (2021).

Photocatalytic study of CeO2-nanocubes supported on palygorskite for ciprofloxacin degradation

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CeO2 is considered a photocatalyst that has a higher degree of crystallinity and larger lateral s, therefore a smaller specific surface area and a lower concentration of oxygen vacancy on the surface [1]. Although this oxide shows photocatalytic activity in the degradation of organic pollutants, certain aspects of CeO2, such as the high recombination rate of electron-hole pairs[2]. To overcome these drawbacks, heterostructures based on clay minerals, such as palygorskite, have been widely used for the removal of various organic pollutants due to their excellent adsorption capacity [2]. The present work is focused on the development of CeO2 nanocubes (CeO2-NC) and its respective hybrid material CeO2-NC/Paly based on the combination of CeO2-NC and palygorskite fibrous clay. These materials were characterized by diverse physicochemical techniques, such as XRD, FTIR, N2 adsorption (BET analysis). To evaluate the photocatalytic activity of the hybrid material, these materials were used employing the CIP antibiotic drug as a contaminant model, and the mechanism of the degradation process was evaluated using the diverse scavenges of the oxide-reduction reaction. The physicochemical characterization pointed out that the structure of CeO2-NC in CeO2-NC /Paly was preserved. The photocatalytic tests showed a total degradation of around 93%, while the isolated semiconductor showed around 60% of ciprofloxacin degradation, indicating that the photocatalytic activity of the CeO2-NC material was improved with the presence of Paly.

References:

[1] PHANICHPHANT, SUKON, AUPPATHAM NAKARUK, AND DUANGDAO CHANNEI. 2016. “Photocatalytic Activity of the Binary Composite CeO 2 /SiO 2 for Degradation of Dye.” Applied Surface Science.

[2] BORGES, LAÍS REIS, ANDERSON GABRIEL MARQUES DA SILVA, ADRIANO HENRIQUE BRAGA, LIANE MARCIA ROSSI, MARCO AURÉLIO SULLER GARCIA, AND PEDRO VIDINHA. 2021. “Towards the Effect of Pt0/Ptδ+ and Ce3+ Species at the Surfaceof CeO2 Crystals:

Photodegradation and photo-Fenton process for removal organic compounds through catalysts ZnO,TiO2, Fe3O4, ZnO-Fe3O4 and TiO2-Fe3O4: a comparative study

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The contamination of the environment by toxic elements due to the increasing anthropic action directly affects the environmental balance. Synthesis and characterization of Fe_3O_4, ZnO and TiO_2[1,2], and nanocomposites system ZnO−$Fe_3O_4$and TiO2−Fe_3O_4 and realized comparative study between photocatalytic processes for Rh B removal were carried out. The nanoparticles were characterized for X-Ray Diffraction (XDR), Scanning Electron Microscope (SEM), Fourier-transform Infrared Spectroscopy (FTIR), Zeta Potential (ZP) and Ultraviolet-visible Spectroscopy (UV-Vis). Results indicate that between photodegradation and photo-Fenton assays there was a variation rate of removal 7.5 to 100% for nanomaterials studed. The presence of light in the Fenton process is favorable to the degradation reaction for magnetic materials, since it is attributed to the reduction of Fe^{3+} to Fe^{2+}, a species that reacts directly with H_2O_2 favoring the Fenton process with formation of hydroxyl radicals, showing an expressive improvement, which is directly related to Fe_3O_4. This theme is current and of great relevance to the synthesis of catalytic materials and environmental issues.

Acknowledgements: Capes, CNPq and POSMAT

References:
[1] MAGDALENA, A. G. ; SILVA, I. M. B. Journal of physics and chemistry of solids, v. 113, p. 5-10, 2018.
[2] SILVEIRA, M.L.D.C. ; DA SILVA, N. R. Research Society and Development, v. 11(2), 2022.

Photodegradation of the drug hydroxychloroquineusing copper-silver titanatenanotubes

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Hydroxychloroquine (HCQ) belongs to the group of 4-aminoquinolines and is used as an antimalarial medication in treating rheumatological and dermatological diseases [1]. In early 2020, during the Covid-19 pandemic, even though it proved to be ineffective against the SARS-COv-2 virus, it was prescribed by medical organizations for the treatment of coronavirus patients [2]. As a result, it has become an organic pollutant in aquatic environments. Photodegradation using titanate nanotubes (TNTs) sensitized with metal ions has become a promising alternative for the remediation of these pollutants. The present research synthesized titanate nanotubes (TNTs) by the alkaline hydrothermal method, then proceeded with ion exchange with $Ag^+$and Cu^{2+}ions to form the Ag_1Cu_{x-1}TNT nanocomposite for application in the photodegradation of HCQ. The prepared materials were characterized by XRD, MET, BET and DRS. The results indicate that $Ag^+$and Cu^{2+}ions are decorating the surface (as Ag and Cu or CuO) and in the interlamellar spaces (as $Ag^+$and Cu^{2+}) of TiO6. In the photodegradation of hydroxychloroquine, the results show that 5.10^{-3}g of the Ag_1 Cu_{x-1} TNT catalyst reaching 81.16% of photodegradation efficiency in a hydroxychloroquine solution of 5.10^{-3}g/L in 120 min under UV-vis radiation. Thus, the Ag_1Cu_{x-1}TNT catalyst is a potential candidate for removing organic pollutants in aqueous solutions.

Acknowledgements
Federal University of Piaui
Federal Institute of Maranhão - Bacabal Campus

References
[1] M. C. Rath, S. J. Keny, H. P. Upadhyaya, and S. Adhikari, “Free radical induced degradation and computational studies of hydroxychloroquine in aqueous solution,” Radiation Physics and Chemistry, vol. 206, no. September 2022, p. 110785, 2023, doi: 10.1016/j.radphyschem.2023.110785.
[2] D. Dabić, S. Babić, and I. Škorić, “The role of photodegradation in the environmental fateof hydroxychloroquine,” Chemosphere, vol. 230, pp. 268-277, 2019, doi: 10.1016/j.chemosphere.2019.05.032.

Photoelectrocatalytic Activity of W-doped Alkali Niobate-Based Perovskite Prepared by Ultrasonic SprayPirolysis

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The photoelectrocatalytic oxygen evolution reaction was studied under visible-light irradiated W-doped niobate-based perovskites. Nanostructured K0.5Na0.5(WxNb1-x)O3 materials were prepared by ultrasonic spray pyrolysis as a function of 1 - 3 mol% W6+ cations within the host structure. A careful characterization of solids including morphology, structure, texture, optical, and electrochemical properties was performed. HRTEM, ED, and XRD characterization showed the KNN structure is distorted by tungsten doping on corner-shared NbO6. XRD analysis showed an expansion of the KNN unit cell due to the difference in ionic radii between Nb5+ and W6+. To the best of our knowledge, the present W-doped niobate-based perovskites showed the highest turnover frequency reported in the photoelectrocatalytic OER under similar overpotential conditions. From the TOF data obtained for the OER, it can be concluded the present W-doped Niobate-based perovskites showed the highest TOF (84.6 s-1) reported up to now for this reaction under visible light irradiation and using similar overpotential conditions. Overall, the present study strengthens the eco-friendly synthesis conditions by ultrasonic spray pyrolysis, being highly favorable as compared to traditional synthesis routes such as high-energy ball-milling [1] and sol-gel [2]. Accordingly, this new class of perovskite-based crystalline materials opens a door for the scaling-up of catalytic and photocatalytic processes related to energy production.

Acknowledgement: CAPES, FAPESP and CNPq.

References

[1] J. Matos, S. Lanfredi, R. Montaña, M.A.L. Nobre, M.C.F. de Córdoba, C.O. Ania, J. Colloid Interf. Sci. 2017, 496, 211-221.

[2] L. Wang, H. Gu, J. He, T. Zhao, X. Zhang, C. Xiao, H. Liu, X. Zhang, Y. Li, J. Alloy. Compd. 2017, 695, 599-606.

Photoelectrocatalytic reduction of CO_2 using $CuBi_2O_4/Cu_2O$ heterostructures for the formation of methanol and methyl acetate

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Using electrochemical (chronoamperometry and cyclic voltammetry) and hydrothermal techniques, $CuBi_2O_4/Cu_2O$ films supported on FTO (fluorine-doped tin oxide) conductive glass were synthesized. The first part is the electrodeposition of a layer of Bi_2Se_3, to form $Bi_2O_3$by heat treatment, then a layer of metallic copper is electrodeposited to transform the structure by applying a temperature of 600 ºC and form $CuBi_2O_4$-CuO, later a layer of $Cu_2O$is electrodeposited on the surface in a basic medium[1] to form $CuBi_2O_4/Cu_2O$ (seeds) and finally it is made a hydrothermal treatment to form the $CuBi_2O_4/Cu_2O$ films. The CO2 reduction was carried out in aqueous solution of Na_2SO_4, $NaHCO_3$, $NaHCO_3/Na_2CO_3$buffer and CH_3COONa as supporting electrolytes, using previously synthesized films as working electrode, Ag/AgCl reference electrode and DSA (dimensionally stable anode) counter electrode. A hermetic sealed reactor (1 atm pressure) was used, which had a quartz window for the passage of light.Work was carried out with different pH (4.15, 7 and 10), the applied potential was also varied (0.0 V, -0.2V and -0.4V) and different electrodes (different charges (Coulumb) of electrodeposition of bismuth) obtaining that the best stability of the material and concentration of methanol obtained (using Gas chromatography for its detection) is at pH 4.15 , potential of 0.0 V and an electrolyte solution of sodium sulfate. research was also carried out to obtain a new product from the reaction of sodium acetate and $CO_2$for the formation of methyl acetate (CH_3COOCH_3), determining the new product obtained from a GC-MS, the variation of potential and pH to obtain a better production of this material, obtaining an improvement at pH 5 and potential of 0.0 V .

Reference

[1]Aranda-Aguirre, A. et al. Photoelectrodes of Cu_2O with interfacial structure of topological insulator Bi_2Se_3contributes to selective photoelectrocatalytic reduction of CO_2 towards methanol. J. CO2 Util. 39, 101154 (2020).

Preparation of activated carbon/TiO_2 hybrid materialfor sulfamethazine photodegradation

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Water contamination with emergent contaminants is a serious issue that can devastate the environment and public health. Thus, developing new and efficient materials for eliminating pollutants is necessary. Activated carbon (AC)/TiO_2, also known as hybrid material, is a compound used in photocatalysis applications to degrade emergent contaminants. Both activated carbon and $TiO_2$are cost-effective, green technologies that can be used to remove pollutants from the environment. This work proposes to obtain activated carbon (AC) with high porosity from crude glycerin by activation with $CaCl_2$as activating agent at 0,005 % to 1% of Calcium (% in relation to polymerized glycerin). The produced AC from crude glycerin and commercial AC are used as substrates for preparing photocatalysts with the impregnating a thin film of a semiconductor, TiO_2, to obtain a synergy effect between the adsorption capacity of AC and the photocatalyst efficiency of TiO_2. The proportion AC/$TiO_2$was a variety (10 e 90) % to specify the material''s efficiency. The specific surface area (S_{BET}) found for AC/90$TiO_2$was 120 $m^2.g^{-1}$. The SEM-EDS micrographs of the materials revealed the existence of carbon, oxygen, and titanium on the surface. The XRD and Raman spectroscopy characterization showed the anatase phase corresponding to the TiO_2dispersed and amorphous phase of AC. The efficiency of materials was evaluated in the photocatalysis of organic contaminants, using sulfamethazine (10 ppm) as a model compound. The results showed the total remotion at 240 min by AC-90TiO_2. In preliminary tests for the AC from glycerin (Gli/0,5%Ca), high adsorption of the sulfamethazine was obtained, about 40%. Thus the preparation of hybrid materials showed efficiency in the organic contaminants remediation by synergetic effects of adsorption and photocatalysis. AcknowledgmentsThis work was supported by CAPES and FAPESP (2019/13471-8).

Probing the Iodine to Bromide phase Photoconversionin Colloidal Thin Film of CsPbBrxI(3-x) nanocrystals

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Perovskite nanocrystals (ABX3) are extremely promising for applications in solar cells, LEDs, photocatalysts, humidity and temperature sensors, memory devices, and low-cost photodetectors. Such characteristics are due to the high fluorescence quantum efficiency allied to the ionic conduction of the charge carriers. However, photoexcitation triggers the ionic diffusion mechanisms and the energy transfer between nanocrystals (NCs), which are poorly understood. [1,2] Thus, halide perovskite NCs with different chemical compositions have been proposed from a theoretical and experimental point of view. Herein, we used hyperspectral fluorescence microspectroscopy to further our understanding of a few optical phenomena in CsPbBrxI(3-x) thin films. We show that CsPbBr2I, CsPbBr1.5I1.5 and CsPbBrI2 nanocrystals thin films samples exhibited conversion from iodide to bromide phases. Initially, the iodide phase undergoes an abrupt drop until stabilization, while the bromide phase begins a significant growth, surpassing the initial fluorescence intensity presented by the iodide phase. We spatially mapped the fluorescence photoconversion and determined their microscale length. A theoretical model was proposed to explain the results.

[1] CHO, Junsang; KAMAT, Prashant V.. Photoinduced Phase Segregation in Mixed Halide Perovskites: Thermodynamic and Kinetic Aspects of Cl-Br Segregation. Advanced Optical Materials, Wiley Online Library, v. 9, n. 18, p. 2001440. 2021. DOI: https://doi.org/10.1002/adom.202001440.

[2] CHEN, Weijian et al. Long-Distance Ionic Diffusion in Cesium Lead Mixed Halide Perovskite Induced by Focused Illumination. Chemistry Of Materials, American Chemical Society, v. 31, n. 21, p. 9049-9056. 2019. DOI: http://dx.doi.org/10.1021/acs.chemmater.9b03320.

Acknowledgments FAPEMIG (BPD-00434-22), CAPES and CNPq.

Production of pure phase mayenite ($Ca_{12}Al_{14}O_{33}$) powders by solution combustion synthesis by varyingfuel and calcination temperature

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This work presents the influence of fuel and calcination temperature to obtain mayenite by solution combustion synthesis (SCS). This is material has characteristics that enable its application as electrochemical components of solid oxide fuel cells, hydrogen storage, cement hydration, among others [1]. All fuels (citric acid, glycine, urea and sucrose) favoured the formation of mayenite, however, only the sample synthesized with glycine (caG) showed 100% of this phase at all three calcination temperatures investigated (1100, 1200 and 1300°C). For the other samples, heat treatment at higher temperatures proves tobe fundamental to obtain the pure mayenite, except for the sample synthesized with urea. Regardless of the fuel used in the synthesis and the phase obtained, the crystallite size, calculated by the Scherrer equation, is smaller than 100 nm, but the caG sample kept the crystallite value (~41 nm) practically unchanged after increasing the calcination temperature. Due to glycine based SCS proved to be more efficient, other analyses were performed only for these powders. From Raman spectroscopy, vibrational structures related to the mayenite phase were identified, which are in agreement with the XRD data for all calcination temperatures. The specific surface area decreased ~19% and laser granulometry analysis revealed an increase of ~4% in the average particle size of this sample (caG) with increasing calcination temperature. Based on the results of this study, it can be concluded that the type of fuel has an influence on obtaining pure mayenite, as well as the calcination temperature and glycine is the fuel that made possible to obtain pure mayenite at 1100°C, that is, at the lowest temperature among those studied.

Acknowledgements: CNPq and CAPES by financial support for the development of research.

Reference:

[1] A. Intiso, F. Rossi, A. Proto, R. Cucciniello, Rendiconti Lincei. Scienze Fisiche e Naturali, 32, 699 (2021)

Raman spectroscopy applied on the structural characterization of TiO_2/expanded graphite films

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Photocatalytic heterojunctions obtained from the use of titanium dioxide (TiO_2) and carbon-based materials are a promising way for the efficient water treatment [1]. The structural properties of the composite photocatalysts are an important characteristic that can influence their photocatalytic behavior [2]. Here, TiO_2/expanded graphite (TiO_2-EG) films were synthetized by sol-gel and deposited on borosilicate glass substrates by airbrush spray coating method at room temperature. Then, the hybrid films were heat treated at 350, 450 and 550 °C. Raman spectroscopy technique was applied to evaluate the effect of carbon amount and temperature on the structural properties of the films. The films heat treated at 350 °C exhibited a characteristic profile of amorphous material. Raman spectra of composite films heat treated at 450 and 550 °C showed well-defined peaks that can be attributed to anatase-TiO_2phase. No peaks related to the rutile or other phases were observed. The investigation revealed that the G and 2D bands present a slight shift, as well as asymmetry, as the carbon content and heat treatment temperature increase - behavior that may be associated with the formation of the semiconductor-C heterojunction. Peaks deconvolution process demonstrated the presence of a second signal not found in the pure films. This effect may indicate a reduction of sp^2domains after the formation of semiconductor-C heterojunction, mainly due to the removal of oxygenated groups during heat treatment, and consequent Ti-C / Ti-O-C bonds formation [3]. The results suggested that the carbon amount and the temperature of heat treatment have great influence on the TiO_2 -EG structural properties, which may contribute to the improvement of the photocatalytic activity of the composite films under visible light.

References:
[1] R.T. Bento et al., Environmental Technology 44, 2023.
[2] R.T. Bento et al., Materials Research Bulletin 143, 111460, 2021.
[3] G. Jiang et al., Carbon 49, 2693-2701, 2011.

Recycling of lignocellulosic waste to prepare hybrid materials for paracetamol removal

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In this work, activated carbons (ACs) were prepared from lignocellulosic residues and used as support for photocatalyst impregnation titanium dioxide (TiO_2). Besides, the efficiency of this material was evaluated in the photodegradation of organic contaminants investigated. AC''s were prepared using the coffee husks, coffee grounds, and sugar cane bagasse as precursors and phosphoric acid as activating agents. $TiO_2$was prepared by the sol-gel method, with hydrogen peroxide (H_2O_2) and without H_2O_2during the synthesis. Then, all the materials obtained were calcined at 350 and 450 °C for 2 h with a synthetic air flow of 100 $mL.min^{-1}$. The materials were characterized according to their physicochemical properties using different techniques. The results obtained from nitrogen adsorption/desorption verified that the specific surface area (S_{BET}) for all prepared AC''s was similar to or higher than the commercial (1000 $m^2.g^{-1}$). The values found were 990, 1344, and 1465 $m^2.g^{-1}$for AC prepared from coffee husks, sugarcane bagasse, and coffee grounds, respectively. X-ray diffractometry (XRD) analysis verified that all TiO_2samples showed peaks referring to the anatase phase. Furthermore, the photocatalytic efficiency of materials was evaluated in the degradation of the contaminant Acetaminophen (Paracetamol) in the aqueous medium. The results showed that TiO_2prepared in the presence of H_2O_2has greater photocatalytic efficiency, removing approximately 94.3% of paracetamol after 300 min. The different TiO_2samples have been impregnated in the AC with the highest surface area (coffee grounds) using a proportion of 15% TiO_2(w/w). The formed composite will be evaluated in the degradation of Acetaminophen.

Role of Tungsten in the Structure and Photoactivity of Alkali Niobates

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Sodium-potassium niobate has been considered a promising eco-friendly ferroelectric material alternative to lead-based compounds [1]. On the other hand, its application in Advanced Oxidative Processes, especially those concerned the photocatalytic reaction, just gained focus recently [2,3]. This work investigated the structure and photoactivity contributions of tungsten doping into the B-site of the $Na_{0.5}K_{0.5}NbO_3$. Nanostructured hollow spheres of $Na_{0.5}K_{0.5}(W_xNb_{1-x})O_3$, where x= 0.01; 0.02; and 0.03 mol% W, were obtained by spray pyrolysis. The powders were produced using a precursor solution (50 mmol/L), which was nebulized at 1.7 MHz and injected by air pumped at 5 L/min into the pyrolytic chamber at 973.15 K. Morphological and structural analysis by Transmission Electron Microscopy and X-ray Diffraction, respectively, confirmed the obtention of the hollow spheres and the perovskite structure. Chemical bonds, band gap energy, and BET surface area were obtained by Infrared spectroscopy, UV-Vis spectroscopy, and N_2-physisorption, respectively. The photoactivity of W-doped KNN was evaluated by means of Basic Blue 41 dye (1L, 26 μmol/L) degradation under UV (7 W, 254 nm) irradiation. The more niobium was displaced by W-doped, the more the photoactivity of the KNN-based materials. The photoactivity increased by up to 14%. The structure and photoactivity of KNN and their relation with tungsten doping are discussed.

Acknowledgments

CAPES (Project 001); FAPESP (2007/03510-9, 2014/11189-0, 2019/06623-6, 2020/00781-6); and LNNano/CNPEM (TEM-C1-20210387).

References

[1] S.K. Mishra, N.S. Choudhury, L. Chaplot, P.S.R. Krishna, R. Mittal, Rev. B: Condens. Matter Mater. Phys. 76, 024110 (2007).

[2] F.R. Praxedes, M.A.L Nobre, P.S. Poon, J. Matos, S. Lanfredi, Appl. Catal. B: Environ. 298, 120502 (2021).

[3] F.R. Praxedes, M.A.L Nobre, A. Olean-Oliveira, M.L. Portugal, P.S. Poon, M.F.S. Teixeira, S. Lanfredi, J. Matos, Appl. Catal. A: Gen. 659, 119171 (2023).

ROS-mediated antibacterial evaluation using TiO2-gum

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TiO2 structures were synthesized by the sol-gel method using Karaya Gum (GKT), having their structure characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-rays (EDS), infrared spectroscopy by Fourier transform (FTIR) and diffuse reflectance and surface area identified by the BET method.The study evaluated the antibacterial activity of GKT against the standard strains of Gram-positive Staphylococcus aureus ATCC 25923 (SA) and Gram-negative Escherichia coli ATCC 25922 (EC). The photoinactivation of bacteria were examined in the dark and in the presence of UV-Vis light - LED system for 2h and the inactivation of bacteria by each test solution was cited. According to the analytical and morphological analyses, GKT was successfully stabilized by the crystalline structure and the presence of TiO2 was elucidated. In testes, GKT showed an inhibitory antibacterial effect of about 20% for S. aureus and 29% for E. coli without the presence of light, and 40% against S. aureus and 70% against E. coli when irradiated by UV-Vis. Bacteria irradiated by light did not follow bacterial inactivation, demonstrating that the lamp used did not influence the process of destruction of the bacterial cell membrane. Furthermore, the composite exhibited the anatase phase of the matrix to thus develop reactive oxygen species (ROS) responsible for bacterial inactivation, transmission of membranes, proteins, lipids and genetic material. Therefore, composite GKT is a promising material for antibacterial applications.

Acknowledgements:The authors would like to thank CAPES, CNPq and FAPEPI.

Saponite-based nanocomposite as an antibacterialagent

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Clay-based nanocomposites are extensively investigated for their properties and diverse applications [1]. When clay minerals are functionalized with metallic oxides, they improve properties such as surface area and chemical and physical stability [2,3]. The present study evaluated the phytostabilization capacity of the ciprofloxacin when adsorbed on saponite pillared with Zr. The results of the nitrogen adsorption/desorption analyses showed a considerable increase in the surface area of the saponite after the incorporation of zirconia. Infrared spectra showed that the drug was effectively adsorbed on the material. The evaluation of the antibacterial activity of the hybrid - before and after the irradiation of the sample for 200 h (lamp 160 W, Hg) - shows the inhibitory effect on the colonies of S. aureus and E. coli. The nanostructured hybrids based on modified saponite are promising materials for biological applications or other approaches.

Acknowledgments: CAPES, CNPq, FAPEPI

REFERENCES

[1] Ruiz-Hitzky, E.; Aranda, P.; Darder, M.; Rytwo, J. Mater. Chem 20, 9306 (2010).

[2] Trigueiro, P, et al. When Dye. Pigment 159, 384-394 (2018). [3] Damacena, D.H.L, et al. Environ. Process 10, 1-24 (2023).

Simultaneous adsorption and photocatalytic removal of tetracycline by MOF-derived ceria

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Water contamination with tetracyclines (TCs), which negatively affects the aquatic biota and the human health, has caused public concern and attracted scientific interest[1]. CeO2 has been one of the most reported lanthanide oxides in catalysis, it is usually studied as a adsorbent and photocatalyst for removing dyes and heavy metals[2]. A pyrolysis at low temperatures was used to transform a metal-organic framework of cerium and benzene-1,3,5-dicarboxylate to mesoporous nanostructured CeO2[3]. The confirm MOF formation and phase purity, PXRD, FTIR and TGA were carried out. TC solutions were submitted to a Fish Embryo Toxicity (FET) to evaluate the toxicity. The characterization confirmed the formation the material. The resulting particles have high capacity for adsorbing TCs from aqueous solution: 90 mg g-1 for 60 mg L-1 TCs. As a result, 98,75% of the initial TC concentration can be removed under simulated sunlight irradiation. According to kinetic analysis, the adsorption of TC onto the particles follows the Freundlich model and is a pseudo-second-order process. Thermodynamic analysis reveals that TC adsorption is exothermic and spontaneous. The embryotoxicity assessment on zebrafish embryos revealed that treatment with CeO2 particles significantly decreased the toxicity of TC solutions. The current contribution highlights attention to the significance of CeO2 in perspective of its interaction with their biological systems, revealing a competitive material that can be applied to real-world situations. We also note that the high surface area CeO2 that is reported here may also have properties suitable for application in other heterogeneous catalysis applications.

Acknowledgements:CNPq, FACEPE, FAPESP and UFRRJ for supporting this work.

References:

[1] S.A. Kraemer, A. Ramachandran, G.G. Perron, Microorganisms 7 (2019).

[2] M. Boaro, S. Colussi, A. Trovarelli, Front. Chem. 7 (2019)

[3] F. PAquin, J. Rivnay, A. Salleo, N. Stingelin, C. Silva, J. Mater. Chem. C. 3 (2015).

SnO2/CuO heterojunctions in methylene blue photodegradation using simulated sunlight

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Scientific interest in the modulation of p-n-type semiconductor heterojunctions has grown, as these materials have shown promising results for photocatalytic applications [1]. Here it is reported that p-n heterojunctions can be applied in the photodegradation of methylene blue (MB). Thus, SnO2, CuO, SnO2/CuO materials with different Cu contents (5, 15 and 25%) were synthed by the precipitation and co-precipitation method. XRD results indicate formation of the rutile phase for SnO2, monoclinic for CuO and for the heterojunctions it was noticed that the addition of Cu caused the loss of crystallinity of the materials [1]. The bandgap energies obtained for the materials were 2.5; 1.4; 2.3; 2.0; 1.5 eV for SnO2, CuO and 5%, 15% and 25% heterojunctions, respectively. Raman spectroscopy showed Ag and Bg vibration modes for CuO and A1g and B1u for SnO2. For heterojunctions, the spectra showed that increasing copper content shifts the intense A1g peak of SnO2 to red, which may be associated with changes in the parameters of the SnO2 network [1]. Furthermore, the appearance of the Ag vibration mode in the SnO2/25% CuO heterojunction can be attributed to the formation of pure CuO species on the surface of the material, which was later confirmed by microscopy images. The reaction results using simulated sunlight revealed that the crystallinity was a determining factor in the activity of the materials, since the SnO2 obtained 55% of MB photodegradation, followed by the heterojunction SnO2/CuO 5% with 53%, the heterojunctions SnO2/CuO 15% and 25% got a reaction result of 37%. Finally, investigating the properties of p-n heterojunctions based on SnO2 and CuO can positively influence the design of efficient materials for photocatalysis induced by simulated sunlight, with potential application in the decontamination of several classes of dyes.

Acknowledgements:

CNPq, CAPES, FAPEAL, UFAL, PPGQB, IQB, GCAR.

References:

[1] Zhou, C. Chemical Engineering Journal. v.359. 2022.

Strategies in the degradation of organic pollutantsusing ZnO-based photocatalysts: A review

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ZnO-based photocatalysts are materials that have excellent performance in removing contaminants due to their good electronic properties and excellent thermal/chemical stability. ZnO presents easy aggregation of particles, non-toxic nature, high surface area, high bandgap and difficult recovery in the reaction medium and, therefore, strategies to immobilize them in different substrates are used to modify the optical performance of photocatalysts from chemical modifications (doping, incorporation, variations in thermodynamic parameters) [1-3]. In the case of doping with metal ions, the bandgap value can be reduced, and its absorption extended to wavelengths in the visible region. In addition, optimized tests are needed on experimental design, synthesis conditions, particle size and structure, and operational parameters (the effect of catalyst amount, the effect of pH, the nature and intensity of light, the effect of oxidizing agent) to improve removal efficiency at low cost [3]. Therefore, this review focuses on recent advances in ZnO-based photocatalysts, such as methods, chemical modifications, choice of organic contaminants in the photocalytic reaction, toxic effects and degradation pathways after effluent release in aquatic matrices, therefore, ZnO is a promising candidate for future research and scientific development.

Acknowledgements:
The authors would like to thank their institutions IFPI, UFPI, UFPB, UFMA, La Plata and USP.

References:
[1] Lum, P.T.; Foo, K.Y.; Zakaria, N.A.; Palaniandy, P. Mater. Chem. Phys., 241, 122405 (2020).
[2] Liu, J.; Luo, Z.; Han, W.; Zhao, Y.; Li, P. Mater. Sci. Semicond. Process., 106, 104761 (2020).
[3] Araujo, F.P.; Trigueiro, P.; Honório, L.M.C.; Furtini, M.B.; Oliveira, D.M.; Almeida, L.C.; Garcia, R.R.P.; Viana, B.C.; Silva-Filho, E.C.; Osajima, J.A. Dalt. Trans., 49, 16394-16403 (2020).

Studies on sinterability and microstructural development of yttrium-doped barium zirconateelectrolytes

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An alternative to lower SOFC temperature is the use of perovskite-type ceramic materials, such as yttrium-doped barium zirconate ($BaZr_xY_{1-x}O_{3-\delta}$, BZY), with protonic conductivity [1]; BZY shows excellent chemical stability and grain conductivity, and finds application not only in SOFC technologies, but also in solid oxide electrolysis cells (SOEC), to produce hydrogen through water splitting [2]. In contrast, it presents poor sinterability, with low densification even at higher sintering temperature the average grain size remains small, and a high grain boundaries area increases resistivity. This behavior is associated to refractoriness but is observed a lack of systematic works to effectively study the cause [3]. In this work, $BaZr_xY_{1-x}O_{3-\delta}$ samples (x=0.2), were produced through solid-state reaction; we were able to obtain samples with 98% of theoretical density after sintering at 1600°C without sintering aid. We proposed a model, based on SEM microstructure and EDS analysis, to the effective reason of poor sinterability, being the formation of a secondary phase at grain boundaries, characterized as an yttrium oxide-rich phase, segregated at boundaries and decreasing the electrical conductivity; electrolytes have a high grain conductivity, but the total conductivity is limited by grain boundaries.

Acknowledgements:

CNPq (process 140451/2014-5), CAPES (finance code 001), UFSCar, UEPG and LCE-UFSCar.

References:

[1] U. Lucia, Renew. Sustain. Energy Rev. 30 (2014) 164-169

[2] J. Y. Y. Huang et al., Int. J. Electrochem. Sci. 18 (2023) 1-5

[3] D. A. Medvedev et al., Prog. Mater. Sci. 75 (2016) 38-79

Study of Cobalt-doped TiO2 Nanostructures for Application in the Photodegradation of Organic Contaminants Via Photocatalysis

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Photocatalysis, which utilizes abundant, clean, and renewable solar energy, is a promising technology for environmental remediation [1]. Titanium dioxide (TiO2) is one of the most promising materials for photocatalysis, but its efficiency is low in the visible spectrum region due to its high bandgap [2]. To improve its efficiency, researchers have searched fornew materials that exhibit good photocatalytic activity in both the ultraviolet (UV) and visible regions. In this study, TiO2 nanoparticles doped with different concentrations ofcobalt ($Ti_{1-x}Co_xO_2$) were synthesized using the modified Pechini method. The samples werecalcined for two hours at a temperature of 500°C, and preliminary characterizations indicate that both the pure and doped samples crystallize in the anatase phase. Furthermore, the morphology of the nanoparticles changes from a more regular pattern inthe pure samples to a more granular one in the Co-doped samples. Photodegradation testsof methylene blue dye using UV light indicate a higher rate of degradation in samples withlower Co concentration. The results suggest that Co-doped TiO2 nanoparticles could bepromising candidates for photocatalytic applications.ACKNOWLEDGEMENTS:FAPESP (2022/02994-20), CNPq (155499/2022-0)REFERENCES:[1] C. Byrne, G. Subramanian, S. C. J. Pillai, Env. Chem. Eng. In Press, 2017.[2] B. Wang, S. Shen, S. S. Mao, J. Materiomics, p.96, 2017.

Study of luminescent nanohybrid of titanate nanotube

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TiO2 is a host candidate for europium ions (Eu3+) to perform metal-semiconductor hybrid due to their applications in optical devices, photocatalysis, and sensors. Here we report the synthesis and characterization of titanate nanotubes (TiNTs) anchored to Eu3+ complex to obtain (TiNTs/Eu(tta)3Phen) nanohybrid, where tta = thenoyltrifluoracetone and phen = phenanthroline, to investigate hybrid structure and photoluminescence properties. TiNTs were synthesized through hydrothermal synthesis and TiNTs/Eu(tta)3Phen hybrid was obtained by dispersing TiNTs in ethanol-SDS solution, followed by adding Eu(NO3)3 and ligands. XRD pattern reveals the anatase phase; from the FT-IR spectrum, it is found that the intensity of the band shifted towards below 1600 cm-1, indicating the formation of nanohybrid. TEM microscopy shows TiNTs are bigger and less fragmented than TiNTs/Eu(tta)3Phen hybrid. The EDS spectrum demonstrates the number of well-defined peaks relative to Ti, O, S, N, and Eu respectively, which support the presence of Ti, O, ligands, and Eu in the nanohybrid. UV-Vis spectrum suggests that the host act as an effective antenna. The photoluminescence spectrum displays a strong red emission corresponding to the 5D0-7F2 transition of Eu3+, in corroboration with fluorescence confocal microscopy images.

References:
[1] LI, Hongbo, et al. Mat. Res. Bulletin, v. 47, p. 4322, 2012.
[2] LI, Hongbo et al. P. tech., v. 212, p. 372, 2011.
[3] WU, Hui-Xia et al. Nanotec., v. 19, p. 345701, 2008.

Study of the adsorption of colors by a nanostructured magnetic material based on graphite and magnetite nanoparticles

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In this study, a magnetic graphite nanocomposite (NGM) based on magnetic nanoparticles (NPs) and graphite nanosheets (GR) was developed as an adsorbent, in order to promote the adsorption of the textile dye reactive black 5 (RB5) from the dispersing medium. NGM was synthed through the hydrothermal synthesis method [1], and the Zero Charge Point (pHzcp) study was performed. For the characterization of NGM and its precursors, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and infrared (IR) spectroscopy techniques were used. An analysis of the stability of RB5 as a function of the pH of the medium was also carried out. The statistical result of the NGM showed a discoloration efficiency (%D) above 90% in the removal of RB5, high adsorption speed, since it reached the adsorption equilibrium in 480 minutes of contact with the adsorbate and presented adjustments greater than 90% for the pseudo first order and second order kinetics, in relation to the adsorption equilibrium was reached around ~45 $mg.g^{-1}$[2]. The study shows that the NGM can be separated from the medium only through the application of an external magnetic field, presenting efficient adsorptive properties, thus showing that it is the main responsible for the removal of the dye.

Synergy between adsorption and photocatalysis: Application of the CuBTC@CuS composite in the decolorization of methylene blue

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Metalorganic frameworks (MOFs) are porous and crystalline solid materials, resulting from the combination of metal ions with organic ligands, forming porous and highly permeable structures. Furthermore, the ability to control the chemical composition and structure of MOFs allows the creation of a wide variety of materials with unique properties and specific applications.[1] The effectiveness of the adsorption capacity of dyes by an adsorbent is influenced by the surface characteristics of the structure and the structural behavior. CuBTC MOF is highly valued due to its large internal surface area and high adsorption capacity, making it economical and safe to remove dyes from aqueous solutions such as methylene blue.[2] This makes the use of MOFs to remove dyes from aqueous solutions an attractive and efficient option. To maximize discoloration, photocatalysis is a technique that can be used to decolorize organic dyes. In this context, copper sulfide (CuS) has been studied as a potential photocatalyst due to its optical and electrical properties.[3] In this work, a new technique of partial modification of the structure of the MOF CuBTC is proposed, resulting in a new compound called CuBTC@CuS. The amount of CuS present in the MOF structure can be controlled by the concentration of HS^- used during synthesis. In the adsorptive process, the composite of CuBTC@CuS was superior to CuBTC, with adsorption of 64% vs. 46%. The composite showed even greater discoloration when irradiated, demonstrating an MB removal efficiency of 87%. Thus, synergistically, the CuBTC@CuS composite showed efficient adsorptive activity and photocatalytic efficiency, which enabled an excellent performance, making the system a promising decolorizer for effluents containing the MB dye.[1] Liu, X. T. et al., Coordin Chem Rev, 476, 214921 (2023).[2] Huang, D. et al., Chemosphere, 263, 127672 (2021).[3] Xiong, Y. et al., Polymers, 11, 1393 (2019).

Synthesis and characterization of Fe(III) and Ga(III) modulated MIL-88B metal-organic frameworks for enhanced stability in aqueous and oxidative environments

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Metal-organic frameworks (MOF) have become increasingly popular due to their unique chemical and structural properties that make them highly desirable for a variety of applications. MIL-88 is a specific family of MOF that consists of Fe(III) ions connected by dicarboxylate ligands, resulting in various pore sizes, shapes, and properties that make them ideal for different uses [1]. However, one major challenge with MIL-88 MOF is their tendency to undergo structural changes or collapse when exposed to water, acidic or oxidative conditions [2]. Hence, this work focused on the synthesis of different Fe1-xGax-MIL-88B and on the evaluation of structural parameters in harmful environments. The MIL-88B were synthesized similarly to methods in the literature [3]. The synthesis were made by modulating the precursor metal salts, FeCl3.6H2O (1-x mmol) and Ga(NO3)3.XH2O (x mmol), and used terephthalic acid as ligand. The reagents were mixed in DMF and NaOH was used as a modulator. The system was heated at 100 °C for 12 h. After cooling, the MOF was washed with DMF and EtOH, collected by centrifugation, and dried overnight at 110 °C. To evaluate the material's stability, tests of soaking MOF in water for 7 d and in H2O2 (0.65 M) at ambient temperature were made. Accordingly to FAAS and XRD, only Fe and Ga-MIL-88B showed iron and gallium leaching in the liquid phase, respectively, which correlates with the diffractograms before and after the tests. Thus, the synergistic effect between iron and gallium proved to be an effective strategy for stabilizing metal ions within the crystalline structure. In future studies, these materials will be evaluated in catalytic reactions and if the modulation of Fe(III) and Ga(III) also enhances others properties. Acknowledgements:FAPESP (N° 2018/01258-5 - 2021/10885-6). References:[1] J. Liu, et. al. Chinese J. Catal. (2021), 42, 12, 2254.[2] S. Jiang, et al. Surf. Interfaces (2022), 30, 101843. [3] S. Choi, et. al. Nanoscale (2016), 8, 37, 16743.

Synthesis and Characterization of g-C_3N_4/C-dot forOrganic Compound Degradation under Sunlight

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The availability and quality of water is one of the great challenges of the 21st century. Searching for water reuse alternative is a topic of first interest. In this work, the low cost and metal free g-C_3N_4/C-dot heterojunction was synthesized aiming its use as visible driven photocatalyst. The precursors, C-dot and g-C_3N_4, were prepared by hydrothermal and thermal heating respectively [1][2]. The g-C_3N_4/C-dot samples were also synthesized by hydrothermal methodologies [3]. The FTIR spectra showed that g-C_3N_4/C-dot showed peaks characteristics of heptazine rings, and a band gap of 2,78 eV were obtained. The fluorescence spectra showed a shift towards the visible region. The efficiency of g-C_3N_4/C-dot was evaluated using a solution of methylene blue at different concentrations. After 180 minutes under sunlight exposure of the solution with heterojunction presence, a discoloration of the solution was observed, indicating a favorable sign for its use in photodegradation.

References:

[1] Lai, X., Liu, C., He, H., Li, J., Wang, L., Long, Q., Zhang, P., Huang, Y. (2020). Hydrothermal synthesis and characterization of nitrogen-doped fluorescent carbon quantum dots from citric acid and urea. Ferroelectrics, 566(1), 116-123.

[2] Maeda, K., An, D., Kuriki, R., Lu, D., Ishitani, O. (2018). Graphitic carbon nitride prepared from urea as a photocatalyst for visible-light carbon dioxide reduction with the aid of a mononuclear ruthenium(II) complex. Beilstein Journal of Organic Chemistry, 14, 1806-1812.

[3] Lv, S., Li, Y., Zhang, K., Lin, Z., Tang, D. (2017). Carbon Dots/g-C 3 N 4 Nanoheterostructures-Based Signal-Generation Tags for Photoelectrochemical Immunoassay of Cancer Biomarkers Coupling with Copper Nanoclusters. ACS Applied Materials Interfaces, 9(44), 38336-38343.

Synthesis and characterization of photocatalyzers SnO_2, NiO and SnO_2/NiO-5%, applied for degradiation of the potential organic pollutant methylene blue

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The present of this work was to investigate the $SnO_2$and NiO photocatalysts, in addition to the SnO_2/NiO-5 % heterojunction, which were synthed by the precipitation and co-precipitation method. The surface, electronic and structural properties were determined by X-Ray Diffraction (XRD), Diffuse Reflectance Spectroscopy (DRS/UV-vis), Infrared Absorption Spectroscopy (FTIR) and N_2physisorption analysis using the method B.E.T. The photocatalytic activity of the systems was evaluated through the photodegradation of methylene blue, a potential organic environmental pollutant. A bandgap value of 2,38 eV for SnO_2, 3,37 eV for NiO and 2,0 eV for SnO_2/NiO-5% was observed, noting that the bandgap for the heterojunction exhibited a decrease compared to the oxides pure, which may be promising for activating this material with wavelengths in the visible range of the electromagnetic spectrum. In addition, the heterojunction presented a surface area value of 62,2 m^2/g^{-1}, higher than the individual. After 120 minutes of reaction in the presence of UV irradiation, the photocatalytic efficiency values of the materials were 54, 32 and 15 % for SnO_2, SnO_2/NiO-5 % and NiO, respectively. The result showed that for $SnO_2$and SnO_2/NiO-5 % heterojunction, which are materials with similar properties, there was a greater efficiency compared to pure NiO. Also, due to the decrease of the main characteristic band of methylene blue, located at 664 nm, the cleavage path of the chromophore structure was identified as the predominant photodegradation route present in the absorbance spectra of all photocatalysts. In general, it is worth mentioning that the literature presents few reports on the synthesis and use of the aforementioned systems in the photodegradation of contaminants.

ACKNOWLEDGMENTS:

FAPEAL, CNPq, CAPES, INCT-CATÁLISE, GCAR, UFAL]REFERENCE:

[1] D. Blažeka et al., Materials, vol. 13, p. 4357 (2020)

Synthesis and photocatalytic characterization of theCTO-ZnS heterostructure

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In the search for sustainable ways to remedy the problems caused by the disposal of contaminated effluents, it is necessary to produce and adapt new materials. The heterogeneous process of photocatalysis is an allied technology because it's an economicaland ecological solution for the degradation of organic pollutants in water [1].

Calcium titanate (CTO) is known to have excellent photocatalytic performance in the degradation of organic and inorganic pollutants [2]. Zinc sulfide (ZnS) is a semiconductor with a fast generation of electron/hole pairs that lead to a good photocatalytic activity [3].

The CTO-ZnS heterostructure was obtained through the SAM method and SEM images revealed that the CTO consists of mesocrystals assembled from agglomerated microcubes. After the application of a ZnS coating, it was possible to identify the formation of a cloud of particles around parts of the CTO mesocrystals, thus forming the heterostructure.

Photodegration tests on Rhodamine B dye in the presence of CTO, ZnS and CTO-ZnS revealed that the heterostructure led to more extensive photocatalytic degradation (50.5%) in 30 minutes than CTO (10%). Furthermore, the degradation efficiency over CTO-ZnS increased to 95.7% after 180 min, indicating that the ZnS coating served to increase the degree of discoloration over time. These satisfactory results demonstrate that CTO-ZnS is apromising photocatalyst capable of accelerating the degradation of organic dyes, thus contributing to the decontamination of water resources.

Acknowledgments:

CNPQ, CAPES e FAPERGS.

References:

[1] Rodriguez-narvaez, O. M. *et al.*Treatment technologies for emerging contaminants in water: A review. **Chem. Eng. J.**323 (2017) 361-380.

[2] Han, J. *et al.*, Fabrication of CdSe/CaTiO3 nanocomposties in aqueous solution for improved photocatalytic hydrogen production. **Appl. Surf. Sci.**459 (2018) 520-526.

[3] Raubach, C. W. *et al.*Strutural and optical approach of CdS@ZnS core-shell system. **Chem. Phys. Lett.**536 (2012) 96-99.

Synthesis, characterization, and photocatalytic performance of Ag-TiO_2

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In this work, by the Pechini method (Pec), $TiO_2$was obtained, no doping (TiO_2Pec) and silver doping (Ag-TiO_2Pec). Characterization was performed by Raman spectroscopy, x-ray diffraction (XRD), and uv-vis diffuse reflectance spectroscopy (DRS). Photocatalytic performance was analyzed by methylene blue (MB) photodegradation and UV-Vis spectroscopy. By Raman spectroscopy characteristic peaks were observed for TiO_2, close to 142, 200, and 395 cm^{-1}(νTi–O), and 637 cm^{-1}(νTi–O–Ti). The synthesized samples indicated increased intensity and narrowing of the peaks, typical of high crystallinity. Inverse behavior in doped samples indicated the influence of silver on the disorder of thecrystal lattice. In the regions, 142 cm^{-1}- 150 $cm^{-1}$and 195 cm^{-1}- 201 cm^{-1}, shifts of 8 cm and 6 cm were estimated, respectively, indicating the presence of silver. Wider bands can also indicate oxygen vacancies [1]. Proportions (anatase:rutile)% were estimated by XRD: TiO_2 Evonik®(72:28); TiO_2 Pec (89:11); Ag-TiO_2 Pec (37:63). The crystallite size (nm): TiO_2Evonik® (22.66); TiO_2Pec (13.71); Ag-TiO_2Pec (17.03) and lattice parameters (a=b≠c) (anatase; rutile) Å: TiO_2Evonik®(3.78≠9.53; 4.58≠2.95); TiO_2Pec (3.78≠9.53; 4.59≠2.96); Ag-TiO_2Pec (3.78≠9.53; 4.6≠2.95). Doped samples showed an increase in peak width and intensity ($\theta \approx 25$ °), and inversion of crystalline phases, predominantlyamorphous system [2]. By DRS, $\lambda_{max}$of absorption (nm) and band gap (E_g) eV: TiO_2Evonik®(358; 3.46); TiO_2Pec (393; 3.14). Photodegradation tests indicated discoloration of MB%: TiO_2Evonik® (100); TiO_2Pec (56.02); Ag-TiO_2Pec (35.84 %). Acknowledgments: Capes, CNPQ, FINEP, UFOB, C-Nano (UNB), and Evonik®Brazil; References: [1] Santos, L. M.; et al.; RSC Advances, 5, 103752 (2015). [2] Akhavan, O.; et al.; J. Materials Chemistry, 20, 7386 - 7392 (2010).

Synthesis, characterization, and photocatalytic performance of C-TiO$_2$

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In this work, by the sol-gel method (SG), TiO$_2$was obtained, without doping (TiO$_2$SG) and doping with carbon (C-TiO$_2$SG). Characterization was performed by Raman spectroscopy, x-ray diffraction (XRD), and uv-vis diffuse reflectance spectroscopy (DRS). Photocatalytic performance was analyzed by methylene blue (MB) photodegradation and uv-vis spectroscopy. By Raman spectroscopy characteristic peaks were observed for TiO$_2$, around 142, 200, and 395 cm^{-1}(νTi–O), and 637 cm^{-1}(νTi–O–Ti). The synthesized samples indicated increased intensity and narrowing of the peaks, typical of high crystallinity. Displacements in the range of 3 to 10 cm^{-1}, and a band around 800 cm^{-1}(C-TiO$_2$SG), indicated the presence of graphitic carbon (C) or carbon replacing oxygen atoms in TiO$_2$surface sites [1]. Proportions (anatase:rutile)% were estimated by XRD: TiO$_2$Evonik® (72:28); TiO$_2$SG (100:0); C-TiO$_2$SG (100:0). The crystallite size (nm): TiO$_2$Evonik®(22.66); TiO$_2$SG (10.03); C-TiO$_2$SG (7.95) and lattice parameters ($a=b\neq c$) (anatase;rutile)Å: TiO$_2$Evonik®(3.79≠9.54; 4.58≠2.95); TiO$_2$SG (3.77≠9.50); C-TiO$_2$SG (3.73≠9.37). Doped samples indicated broadening and reduction of peak intensity and size of smaller crystallites, indicating that carbon atoms may have influenced crystal growth and an amorphous system [2]. By DRS, $\lambda_{max}$of absorption (nm) and band gap (E_g) eV were estimated: TiO$_2$Evonik®(358; 3.46); TiO$_2$SG (371; 3.34); C-TiO$_2$SG (380; 3.26). Photodegradation tests indicated discoloration of MB%: TiO$_2$Evonik®(100); TiO$_2$SG (75.73); C-TiO$_2$SG (73.53). Acknowledgments Capes, CNPQ, FINEP, UFOB, C-Nano UNB, and Evonik®Brazil. References [1] Taziwa, R.; et al.; J. Nanoscience Nanotechnology Research 1, 1-11 (2017).[2] Akhavan, O.; et al.; J. Materials Chemistry 20, 7386 - 7392 (2010).

Synthesis of a hybrid of TiO_2 and graphene derivedfrom biomass

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Titanium dioxide (TiO_2) is a widely used photocatalyst, but it has some limitations that restrict its use, such as a high bandgap value (3.2 eV), which makes it inactive in visible light. $TiO_2$is often modified by the addition of carbon nanomaterials to form a hybrid material to improve its efficiency with visible light and eliminate the recombination of photogenerated electron holes. Graphene-based materials can be attached to TiO_2, resulting in synergic properties and increasing the functionality of the hybrid [1]. This hybrid material could be the future of photocatalysis, particularly in pollution remediation and, energy generation using sunlight [2]. The main aim of this study is to produce a hybrid material of $TiO_2$and graphene derived from biomass capable of degrading pollutants and generating hydrogen gas (H_2) through water splitting under visible light.

Acknowledgments: Anna Luísa Miguel thanks Coordenação de Aperfeiçoamento de Pessoalde Nível Superior - Brasil (CAPES) - Finance Code 001. The authors thank Evonik forproviding the P25 samples. Roberto de Avillez, and Sonia Letichevsky thank grants fromBrazilian agencies FAPERJ (E-26/202.870/2018, and E-26/010.000982/2019). Roberto Ribeiro de Avillez also thanks grant from CNPq (302776/2022-2).

[1] Purabgol, A., Mayilswamy, N. Kandasubramanian, B. Environ. Sci. Pollut. Res. 29, 32305-32325 (2022)

[2] Nasir, A., Khalid, S., Yasin, T Mazare, A. Energies, 15, 33 (2022).

Synthesis of metal doped TiO_2 nanotubes through one-step anodization for photoelectrochemical water splitting

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The utilization of clean solar energy is essential to provide energy supply for the future. Photoelectrochemical (PEC) water splitting (WS) is one of the most promising technologies for solar energy conversion into renewable fuels [1]. In that sense, TiO_2 has emerged as a strong candidate owing to its excellent stability, low cost, and nontoxicity. Besides, TiO_2 nanotubes (TiO_2NTs) obtained through anodization present an organized and larger surface area when compared to other nanophotocatalysts. Additionally, the thin walls of theNTs lead to a minor chance of charge carrier recombination increasing the WS reaction rate[2]. With anodization, the doping of TiO_2NTs can be accomplished during its synthesis to optimize electronic properties of TiO_2NTs. In this work, highly organized TiO_2NTs wereobtained through an anodization process for WS applications. Polished Ti foils were placedas anodes and Cu as cathodes in a beaker with electrolytes. A potential difference of 20-60 Vwas applied per 2h. The electrolyte contained ethylene glycol, H_2O, NH_4F, and metal salts.Thermal treatment was done at 500°C per 3h to increase crystallinity. Optical and morphological characterizations were done using UV-Visible spectroscopy and scanning electron microscopy (SEM). Photocurrent measurements and the material efficiency datawere obtained with PEC essays in KOH 1 mol L^{-1} using a high-pressure Hg/Xe lamp with 140W maximum power. TiO_2NTs obtained are anatase phase with a bandgap of 3.2 eV. SEM images show NTs of diameters of 40-120 nm. Photocurrent measurements show a 10-time photocurrent density increase after the anodization, reaching 1.6 mA/cm^2 at 1 V. Doped TiO_2NTs obtained through one-step anodization process are underway.

Acknowledgments
The authors thank the Programa de Pós-graduação em Química and the Programa de Formação de Recursos Humanos for the sponsorship.

References
[1] J. Phys. Chem. C (2012), 116, 16, 9049-9053
[2] Ceramics International 47 (2021) 18358-18366

Synthesis of Co-POM@ZnO for purification of water contaminated with organic dyes

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Water pollution due to toxic organic compounds is a serious problem nowadays since the legal regulations concerning disposal of chemicals into the natural environment are more and more restrictive. The main causes of surface water and groundwater contamination are industrial discharges. Polyoxometalates (POMs) are a class of inorganic anionic metallic oxygen cluster compounds with abundant charge and excellent physical and chemical properties, such as photocatalytic activities, strong electron acceptability, adjustable energy band structure, and reversible multi-electron redox properties. Additionally, they are also inexpensive and environmentally friendly. It has been reported that POMs@TiO2 compounds improved activity for the photocatalytic degradation of organic pollutants such as dyes. In this study, photocatalytic oxidation of methyl orange dye under irradiation a Halogen lamp 900 W have been tested over Cobalt ion-based polyoxometalate@ZnO photocatalysts, with different molecular weight. The degradation of methyl orange is up to 97% with Co7@ZnO in 30 min, up to 92% with Co5.5@ZnO and Co4@ZnO about 83% within 120 min, at conditions 6 mg/L concentration of compound and 2 mg/L concentration of methyl orange. Fast degradation was obtained with increasing of the molecular weight of POM photocatalyst. The degradation of MO showed that the combination of ZnO and Co-POM compounds can degrade methyl orange more effectively in aqueous solution.

Synthesis of silver nanostructures for catalytic applications

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Water contamination poses ecological threats and reduces the availability of freshwater. Organic pollutants can resist degradation and cause bioaccumulation, becoming a global concern that needs to be addressed. The treatment of organic pollutants is not always adequate or efficient, so proper and effective remediation procedures need to be developed and implemented. Advancements in nanotechnology have shown that nanoparticles (NPs) present the potential for environmental remediation. NPs possess a high surface area/volume ratio, which can improve the sensitivity and performance of monitoring tools. Among these types of nanomaterials, silver nanoparticles (AgNPs) stand out.[1,2] Thus, in this work, we prepared AgNPs with different shapes for the catalytic degradation of the organic pollutant 4-nitrophenol, in water. The AgNPs were synthesized in an aqueous medium by chemical reduction methodology, using polyvinyl alcohol (PVA) and polyvinylpyrrolidone (PVP) as stabilizers, sodium borohydride as the reducing agent, and hydrogen peroxide for morphological control. The results obtained showed that AgNPs were successfully prepared using polyvinyl alcohol (PVA) and polyvinylpyrrolidone (PVP) with controlled shape, achieving nanospheres or nanoprisms. The prepared AgNPs successfully promoted the reduction of 4-nitrophenol to 4-aminophenol. Thus, AgNPs have the potential to be used as a catalyst for pollutants removal in water.

Acknowledgments:

The authors thank the UFPE, CNPq, FACEPE (IBPG-1071-3.03/22) and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020).

References:[1] Wang et al., Applied Catalysis B: Environmental, 2018, 237, 721.[2] Venieri et al., Sustainability 2020, 12, 10047.

Synthesis of tin niobate (SnNb2O6) by solution combustion, characterization and study of its photocatalytic property.

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Synthesis of tin niobate (SnNb2O6) by solution combustion, characterization and study of its photocatalytic property

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Summary:

The growing human urbanization and industrialization has caused several problems to the environment, mainly related to water resources. In view of this problem, research has been carried out in order to seek solutions to minimize this contamination through photocatalysis, using semiconductors[1]. Among these semiconductors, niobates have been used for this purpose. Tin niobate (SnNb2O6), due to its semiconductor characteristics, crystalline structure and reduction potential, is an excellent material with photocatalytic properties However, due to morphological characteristics such as specific surface area, obtaining it is not so simple. In this sense, the technique of synthesis by combustion in solution will be used because it is a versatile and simple method, in addition it allows obtaining materials with high reaction yield, as well as obtaining powders with high surface area. Chemical processes and the X-Ray Diffraction (XRD) characterization technique that occurred in crystalline phases were carried out. Measurements of photocatalytic properties will be produced in a photocatalysis chamber, using lamps with visible radiation emission directly on the elements to be degraded (Rhodamine B and Methyl Orange in aqueous solution) together with the semiconductor powder[2].

Acknowledgements: Fapema, Ifma.

References:[1] MITCHELL.D1 and ENERMARKB.S2, Journal vol48., page.190-198 (2015)
[2] ZAYED.A1 and ABDEL.E2,Journal vol 49,page.166 (2018)

Synthesis of WO_3/Nb_2O_5 heterostructure applied inorganic dyes removal from aqueous solution

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Water pollution, caused by dyes, is a global issue. Advanced Oxidative Processes have emerged as a viable solution to address this issue, with Heterogeneous Photocatalysis beinga popular process due to the formation of radicals with high oxidation power that can degrade organic compounds [1]. In this study, $Nb_2O_5$and WO_3were coupled to improve the properties for the photocatalytic process. The synthesis of isolated materials was based on previous reports. $Nb_2O_5$was synthesized based on the procedure reported by Leite et al. [2], and $WO_3$was prepared using the procedure described by Castro et al. [3]. For the synthesis of the WO_3/Nb_2O_5heterojunction, acid tungstic was dissolved in hydrogen peroxide (H_2O_2). To this solution was added ammonium niobium oxalate dissolved in distilled water and then $H_2O_2$was added. The volume was completed with distilled water and the solution was treated at 200 ºC for 24 h. Isolated materials were prepared for comparation in the same conditions. The samples were characterized, and the results showed the presence of $WO_3$and Nb_2O_5, indicating the formation of heterojunction. To analyze its photocatalytic properties, the samples were applied in photodegradation of MB and RhB dyes under UV light. The results showed that the heterojunction was more effective than the pure materials. Therefore, WO_3/Nb_2O_5heterojunctions have the potential to be applied in photocatalytic processes for the degradation of organic compounds.

Acknowledgements: The authors thank CBMM, CAPES, Embrapa Instrumentation.

References:

[1] C. Ucker, et al., Chemical Physics Impact 4, 100079 (2022).
[2] E. R. Leite, et al., The Journal of Physical Chemistry B 110, 1088 - 1090 (2006).
[3] I. A. Castro, W. Avansi, C. Ribeiro, Crystal Engineering Communication 16, 1514 - 1524 (2014).

Synthesis of zinc oxide nanosheets for environmental applications

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Zinc oxide nanoparticles (ZnO-NPs) are used as semiconductors in photocatalysis and decontamination of pharmaceutical, textile industry, and domestic effluents. Different NPs morphologies can change the required properties in each application. Synthesis of ZnO-NPs was carried out with a modification of the method proposed by Sharma et al. [1,2]. The volume of 0.1M of $ZnSO_4 . 7H_2O$ was added dropwise (16μL/s) to 0.4M of NaOH (1:4M). The product obtained was washed in a cellulose filter under negative pressure and heated at 60°C for 1h followed by a controlled air atmosphere at 150°C in an electric muffle furnace. Thermal treatment in chemical synthesis was an important factor to convert secondaryphases as $Zn(OH)_2$to ZnO. The diffraction peaks by X-ray diffraction (XRD) match to hexagonal wurtzite structure. The morphology of ZnO nanosheets and particles (21±3nm) was evaluated by images of FE-SEM. The bandgap value for ZnO-NPs was around 2.26 eV. In photocatalysis with UV vis, the absorption of photons leads to the excitation of the electron from the valence band to the conduction band, generating holes in the valence band that can oxidize or reduce effective organic compounds [3]. Thus, the use of ZnO nanosheets in photocatalysis can be efficient in wastewater treatment.

Acknowledgments:

CAPES- Brazil - Finance Code 001 and CNPq grants 309186/2020-0.

References:

[1] D. Sharma, S. Sharma, B.S. Kaith, J. Rajput, M. Kaur, Synthesis of ZnO nanoparticles using surfactant free in-air and microwave method, Appl. Surf. Sci. 257 (2011) 9661-9672

[2] D. Sharma, J. Rajput, B.S. Kaith, M. Kaur, S. Sharma, Synthesis of ZnO nanoparticles and study of their antibacterial and antifungal properties, Thin Solid Films. 519 (2010) 1224-1229

[3] J.R. Tamashiro, I.S. Lima, F.F.G. de Paiva, L.H.P. Silva, D.V.M. de Oliveira, O. Baffa, A. Kinoshita, Treatment of sugarcane vinasse using heterogeneous photocatalysis with zinc oxide nanoparticles, Sustain. 14 (2022)

The impact of synthesis conditions on the photocatalytic behavior of SnO_2 nanoparticles

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Tin dioxide (SnO_2) has been widly investigated and used in different applications, especially in heterogeneous photocatalysis [1]. This work aimed to evaluate the influence of structural characteristics of SnO_2on the degradation process of dyes and drugs. The oxide samples were obtained by the polymeric precursors method and calcined at 700°C for 2, 4 and 6h, and characterized by X-ray diffraction (XRD), Raman spectroscopy, infrared (IR), UV-Visible (UV-vis) spectroscopy, Scanning Electron Microscopy (SEM), BET analysis and Zeta potencial measurements. XRD structural results of SnO_2samples confirmed the formation of rutile-like tetragonal crystalline phase (*P42/mnm*), with na increase of the long-range structural order as indicated by FWHM values and crystallite , showed the influence of calcination time on the structural organization of the materials, indicating greater. The Raman spectra confirmed the presence active modes and acoustic modes in SnO_2. Regarding the photocatalytic properties of the materials, the sample at 700 °C/2h with maximum efficiency of 91% in the decolorization of the cationic crystal violet (CV) dye and 65% in the of the anionic orange gelb (OG) in agreement with the Zeta potencial analysis. In relation to tetracycline antibiotic, 52% of its degradation was attained by SnO_2calcined at 700 °C/2h after 6h of reaction. Additionally, the influence of catalyst mass and dye concentration was studied.

Acknowledgements:

FAPESQ-PB/PRONEX , PRPGP/UEPB, NPE-LACOM/UFPB, CNPq/ MCTIC, FAPESQ-PB/SEECT and CAPES.

References:

[1] Haq, S. ; Rehman, W. ; Waseem, M. ; Shah, A. ; Khan, AR; Rehman, M. ; Ahmad, P. ; Khan, B. ; Ali, G.. Mate. Res. Expres, 7, 025012, 2020.

The influence of structural modifications in zirconate-stannate perovskites on the photocatalytic efficiency of RNL and MG dyes

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ABO3-type mixed oxides have been acting as catalysts in the photodegradation processes of textile dyes, which have been presented as one of the main villains in relation to their impacts on the environment due to their complex treatment when present in water bodies [1]. In this context, the present work aimed to prepared alkaline-earth metals stannates and zirconates for applications in the discoloration of malachite green (MG) and Remazol Yellow Gold (RNL) dyes. The oxides were synthed by the modified pechini method and characterized by X-Ray Diffraction (XRD), Infrared Spectroscopy (IR), Raman and UV-Visible spectroscopy. MSnO3-based perovskites (Ba2+, Ca2+ and Sr2+), BaZr0.5Sn0.5O3 and BaZrO3 were successfully obtained, confirmed by XRD patterns. Raman spectra of stannates and zirconates revealed a lower degree of symmetry according to the modifier cation with the largest ionic radius (Ba2+) and a greater degree of symmetry in BaZr0.5Sn0.5O3. From the UV-Vis spectra, band gap values ranging from 3.25 to 5.01 e.V. were observed. In relation to the photocatalytic tests the BaSnO3 samples showed an efficiency of 88.9% and 91.2% discoloration of the MG and RNL, respectively, in 6 h of reaction. BaZr0.5Sn0.5O3 presented 77.5% efficiency for the VM dye in only 2 h. These results showed that structural order-disorder modified by network modifier cation positively influenced the photocatalytic performance of the materials.

Acknowledgment: UEPB/PRPGP/PPGQ FAPESQ/PRONEX, and NPE-LACOM/UFPB.

[1] Nascimento, JLA and Chantelle, L. et al. Catalysts, 12, 428(2022).

TiO2/Nb nanofibers and photocatalysis in thepetroleum industry: effluent treatment

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Petroleum is one of the main natural resources used as a source of energy today. In the petroleum industry, the refining stage is the most polluting stage, which can contaminate soil, air and water. The high demand for water by refineries actually becomes a problem due to the large discharge of difficult-to-treat liquids into the environment. A significant part of the discarded effluents are composed of water produced from petroleum, which has inorganic salts, oil, grease and organic contaminants. Such components have recalcitrant characteristics and generate water pollution, affecting human health and causing ecological imbalance [1,2]. Phenol, one of the main components of petroleum, is present in water produced from oil and has high toxicity, low biodegradability, in addition to carcinogenic and mutagenic behavior. In view of the problem of effluent disposal, it is necessary to develop solutions so that these effluents are treated in an ecologically correct, sustainable and economical way. Photocatalytic processes are widely used for wastewater treatment, being responsible for the degradation of organic and inorganic contaminants [3]. In this sense, this work aims to produce TiO2 nanofibers doped with niobium in different concentrations as photocatalysts using the electrospinning technique, in addition to evaluating and characterizing the structural, morphological and photocatalytic properties of the synthesized material. The photocatalytic properties will be evaluated from phenol degradation under UV light.

Acknowledgements: LSQM, PPGCEM, UFRN PRH, FINEP, ANP.

References:

[1] M. Almeida, IMPACTOS AMBIENTAIS CAUSADOS PELA INDÚSTRIA PETROLÍFERA, Revista Dissertar. 1, 69-80 (2019).

[2] S. Jiménez, State of the art of produced water treatment, Chemosphere. 192, 186-208 (2018).

[3] D. al deen A. Aljuboury, Optimization of the petroleum wastewater treatment process using TiO2/Zn photocatalyst, S Afr J Chem Eng. 38 (2021).

Tungsten trioxide synthed by the modified-Pechini method and applied as photocatalyst.

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Hydroxyl radicals (·OH) are responsible for photocatalytic oxidation reactions. For this reason, the identification and quantification of this radical becomes an important parameter in the evaluation of the activity and efficiency of a photocatalyst [1]. In the present work, tungsten trioxide (WO3) was synthed by the modified Pechini method, to be used in the photohydroxylation of terephthalic acid (AT), in order to evaluate the formation of ·OH. The material was characterized by X-Ray Diffraction, Infrared Spectroscopy and Raman Spectroscopy and Scanning Electron Microscopy. WO3 is an n-type semiconductor, whose crystalline structure depends on temperature, so the results indicated a tetragonal crystalline structure (α-WO3), after calcination at 800°C for 4 h. The emission spectra were analyzed in a spectrofluorimeter as a function of the reaction time, where the photogeneration of hydroxyl radicals by the semiconductor resulted in the formation of 2-hydroxyterephthalic acid, a highly fluorescent compound. Therefore, the production of ·OH was proportional to the irradiation time of the catalyst suspension with the terephthalic acid solution. The results, when compared to the blank, show the applicability of the WO3 catalyst, in which the high intensities after its use resulted in a high production of hydroxyl radicals, playing an important role for a good photocatalytic efficiency.

Acknowledgements:
CAPES/MEC, PRONEX/CNPq-MCTIC/FAPESQ, FINEP/MCTI, Ed. 018/CNPq/MCTI

References:

[1] ŽERJAV, G. et al. Applied Catalysis A: General, v. 598, p. 117566, 2020.

USE OF CERIUM-CONTAINING ZINC OXIDE FOR PHOTOCATALYTIC APPLICATION

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The pharmaceutical industry is one of the significant waste generators, and pharmaceuticals are among the most commonly assessed emerging contaminants [1]. To minimize the problems caused by persistent organic pollutants, advanced oxidative processes and remarkably heterogeneous photocatalysis have emerged as promising alternatives for degrading and mineralizing these pollutants in wastewater [2]. Photocatalysis using Zinc Oxide containing Ce was proposed to evaluate the efficiency of pharmaceutical degradation.Photocatalytic tests were conducted using different concentrations of the catalyst in suspension with the IBF drug solution, and the toxicity of the solution to Artemia salina wasalso evaluated. The tests analyzed the effect of pH and catalyst concentration over a maximum period of 120 minutes of exposure to UV radiation during predetermined timeintervals. The different reduction efficiencies of IBU were 60%, 46%, and 40% for catalystconcentrations of 0.5 g L^{-1}, 1.0 g L^{-1}, and 1.5 g L^{-1}, respectively. The photocatalytic performance was sensitive to changes in pH, with the acidic condition proving the most efficient, demonstrating good photocatalytic activity with an equivalent rate of 47%. Thetoxicity tests showed low mortality from the irradiated solution.

Acknowledgment: CAPES, CNPq, FAPEPI

References:

[1] MONTEOLIVA-GARCÍA, A., MARTÍN-PASCUAL, J., MUÑÍO, M. M., et al. Removal of carbamazepine, ciprofloxacin, and ibuprofen in real urban wastewater by using light-driven advanced oxidation processes , International Journal of Environmental Science and Technology, v. 16, n. 10, p. 6005-6018, 2019.

[2] HONORIO, L. M. C., TRIGUEIRO, P. A., VIANA, B. C., et al., Nanostructured Materials for the Photocatalytic Degradation of Organic Pollutants in Water . Nanostructured Materials for Treating Aquatic Pollution, [S.l: s.n.], 2019. p. 65-90

Use of iron oxide magnetic nanoparticles in the adsorption of Rhodamine 6G and Rhodamine B dyes

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Rhodamine 6G (Rh6G) and Rhodamine B (RhB) are dyes widely used in industries, such as textiles, inks for printers, and lasers, among others. To minimize the environmental impact from the improper disposal of residues contaminated with these dyes, several types of effluent decontamination processes have been studied. The adsorption method has stood out in the treatment of these effluents, due to its ease of operation, low cost, and high efficiency. In this work, iron oxide nanoparticles (NPs) were synthed by co-precipitation and tested as adsorbents for Rh6G and RhB. The phase of the NPs was determined by XRD to be the cubic Fe3O4, with mean crystallite s around 46.3 nm. Scanning electron microscopy showed spherical morphologies for the NPs, with diameters around 51.0 ± 5.0 nm. The maximum absorption wavelengths for Rh6G and RhB were 526 nm and 554 nm, respectively. Both rhodamines were stable when the pHs of the media were changed, with no absorption changes in their visible spectrum. Adsorption tests of the rhodamines by NPs were carried out. The highest adsorption capacity occurred at pH 4.0 and the adsorption equilibrium at the contact time was close to 30 min. The Pseudo-Second Order model best described the adsorption kinetics, with a 99% adjustment, in comparison with other models. The Temkin model fit very well the adsorption isotherms obtained at 301, 311 and 321 K. The maximum adsorption capacity occurred in both dyes at a temperature of 321 K: 279.51 mg g-1 for Rh6G and 276.64 mg g-1 for RhB. This study shows that the synthed NPs are efficient adsorbents for rhodamines in aqueous medium. Furthermore, the NPs can be easily removed from the medium through the application of an external magnetic field, making this a promising system for the treatment of effluents.

Use of reactive species to understand the behavior of TiO2/Ag photocatalysts

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Despite the known contamination of aquatic environments by different pollutants, cheap efficient treatments for contaminated water are still not a reality. One of the forms of remediation is photocatalysis. In the present study, changes in photocatalytic processes after photodeposition of Ag on the surface of commercial TiO2 were evaluated. One way to evaluate the species active in photocatalysis is by the use of substances that react with the hole, removing them from the solution and reducing the photocatalytic activity, which was done with EDTA addition into the solution [1]. To avoid the reaction of electrons with O2 dissolved in water, the reaction was carried out in a stream of N2. Materials were characterized by X-ray diffraction (XRD), infrared absorption spectroscopy (IR), Raman spectroscopy and scanning electron microscopy (FEG) coupled to EDS. The presence of Ag on the sample was confirmed by the positions of the surface plasmon resonance bands in the UV-vis spectra, besides a color change of the samples. XRD patterns displayed a peak assigned to Ag at 44º. FEG/EDS analysis confirmed the deposition of Ag on the TiO2 surface, while zeta potential indicated a change in the surface charge after Ag deposition, particularly at low pH values. The fluorescence spectrophotometer allowed us to analyze the behavior of the TiO2/Ag composite in the formation of hydroxyl radicals, by the photohydroxylation of terephthalic acid, with EDTA addition or under N2 flow. EDTA addition decreased the amount ·OH radicals, reducing the photocatalytic activity, confirming the participation of holes in the photocatalytic process, for all samples. N2 flow also decreased the photocatalytic activity, which indicates that the process of excitation of electrons to the higher energy band and subsequent reaction with O2 was effective. ACKNOWLEDGMENTS: CAPES, PRONEX/CNPq-MCTIC/FAPESQ, PIBIC/CNPq/UFPB REFERENCES: [1] L. M. C. HONORIO et al. Applied Surface Science 2020, 528, 146991.

Utilization of bimetallic Fe/Co-MOF for dye adsorption

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Contamination of water resources by dyes is one of the most serious ecological threats. Dyes often reveal toxic, carcinogenic, and mutagenic properties [1]. Among water treatment methods, adsorption is one of the most efficient for separating dyes from wastewater [2]. One of the most efficient sorbents for dyes are Metal-Organic Frameworks (MOFs). They are porous coordination polymers, with large specific surface areas, variety of active sites, and affinity to ionic and non-ionic molecules [3]. In this work, the capacity of dye adsorption was evaluated in batch adsorption experiments at room temperature using several dyes (cationic and anionic) and a bimetallic MOF (FeCo-BDC). The FeCo-BDC was characterized by FTIR, PXRD, and SEM-EDS. The FeCo-BDC exhibited a pure crystalline phase and adsorbed over 80% of anionic dyes in only 20 min of phase contact. The excellent adsorption capacity can be attributed to the ionic interaction between the active sites of the MOF groups and the dyes.

Acknowledgments:
The authors would like to thank CAPES (643078/2021-00), CAPES/PRINT (194800/2018-00), and CNPQ (130230/2022-7) for the scholarship grants. And M.A.O gratefully acknowledges the support from Mayor of Gdańsk Daniel Gabriel Fahrenheit Scholarship Program for Students and International PhD Students.

References:
[1] O. J. S. Junior., et al., Mater. Chem. Phys., 235, 121737 (2019).
[2] K. Hemine, N. Łukasik, J. Hazard. Mater., 418 (2021).
[3] B. S. Barros, et al., ChemistrySelect, 3, 7459-7471 (2018).

Zinc imidazolate structure supported on TiO2 nanotubes a promising third-generation photocatalyst

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Zinc imidazolate structure supported on TiO2 nanotubes a promising third-generation photocatalyst
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The compounds formed by imidazole ligands form crystalline structures known as ZIFs (Zeolitic Imidazolate Framework), are materials that have well-defined structures of high stability, crystalline, porous and have a large surface area [1], the obtaining of materials supported on solid substrates, that have as characteristics of acting as potential photocatalysts, promotes what we call third-generation materials [2]. In this work, the material called (ZnIm-NTT), was developed by joining of the zinc-imidazolate framework [Zn2(C3H3N2)4]n (ZnIm) [3] formed in situ on TiO2 nanotubular structures (previously obtained by anodization) via hydrothermal synthesis. The diffractogram of the material produced by XRD shows the obtaining of a high crystalline material, with SEM and EDS analysis it is possible to see the structure deposited on the nanotubular surface and its chemical characterization. The compound was applied as a photocatalyst for the degradation of the persistent organic pollutant (POP), methylene blue dye in the concentration of 15 ppm, via simulated solar radiation, promoting a percentage of color removal of over 96 % after 450 minutes. These results show evidence that the third generation material obtained, here has a potential application in the treatment of persistent organic pollutants.

Acknowledgements:
Work supported by CNPq and Cetene

[1]Zhao, X.; Li, J. Chinese Journal of Catalysis, v. 42, p. 872-903, 2021.
[2]ANWER, H.; MAHMOOD, A.; LEE, J.; KIM, K.; PARK, J.; YIP, A. Nano Research, v. 12, p. 955-972, 2019.
[3]SPENCER, E. C.; ANGEL, R. J. CHEM. SOC., v. 131, p. 4022-4026, 2009.

Zinc molybdate synthed by the Pechini method and evaluated as photocatalyst

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In the last years, transition metal molybdates (AMoO4, A = Cu, Zn, Co, Ni and Ca) have been widely studied due to their structural, electronic and catalytic properties. This versability of catalytic properties are related to the different oxidation states that molybdenum may have. Among these compounds, zinc molybdate has been applied as photocatalyst, with interesting results. In the present research, zinc molybdate was synthed by the modified Pechini method; characterized by X-ray diffraction, infrared spectroscopy, Raman spectroscopy, nitrogen adsorption and scanning electronic microscopy; and applied as photocatalyst in the degradation of methylene blue (MB) and in the photohydroxilation of therephtalic acid (TA). A single-phase material was obtained with triclinic structure, ·-ZnMoO4. Photohydroxilation results indicated that a small amount of hydroxyl radical was formed, indicating that the indirect mechanism is not the main process of photodiscoloration. This result is similar to those obtained for MoO3 by our research group. In this case, photocatalysis may occur by the direct mechanism, with the electron transfer between the photocatalyst surface and MB.

References:
[1] Jing Zhang et al. Chinese Journal of Catalysis 38 (2017) 2009-2020.
[2] Majid Ramezani et al. J Mater Sci: Mater Electron (2015) 26:7588-7594.

ZnO/TiO₂/Ag₂Se nanostructures for photoelectrocatalytic degradation of ciprofloxacin

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The aim of this work is ciprofloxacin (CIP) removal using a $ZnO/TiO_2/Ag_2Se$ compound by photoelectrocatalytic reactions. The material was prepared as follows: first, the ZnO seeds were electrodeposited onto FTO conducting glass, followed by hydrothermal treatment to turn them into nanorod a second process uses the sol-gel method (dip-coating) for TiO_2synthesis and a third, for synthesis and Ag_2Se electrochemical deposition. The XRD diffraction peaks can be indexed as hexagonal wurtzite structure ZnO, the presence of TiO_2in its anatase form and of the Ag_2Se orthorhombic phase. FTIR spectroscopy shows the ZnO, $TiO_2$and Ag_2Se bands at 582, 848 and 1666 cm^{-1}respectively. Raman spectroscopy showed the presence of the TiO_2anatase phase. The SEM-EDS images displayed ZnO nanorods presence coated by TiO_2particles, as well as Ag_2Se coating the $TiO_2$and ZnO surface, as dispersed nanoparticles. Synthetic water containing 5 mg dm^{-3}CIP solution in 0.75 mol L^{-1}NaCl at pH 7 was used to study photoelectrocatalytic behavior of $ZnO/TiO_2/Ag_2Se$ compound, attaining 71% CIP decay after 3 h at bias potential +0.80V vs Ag/AgCl under irradiation with blue LED lamp 440 nm.

References

[1] A. Aranda, R. Landers, P. Carnelli, R. Candal, H. Alarcón, and J. Rodríguez, “Influence of silver electrochemically deposited onto zinc oxide seed nanoparticles on the photoelectrochemical performance of zinc oxide nanorod films,” Nanomaterials and Nanotechnology, vol. 9, May 2019, doi: 10.1177/1847980419844363.

[2] K. Changanaqui, E. Brillas, H. Alarcón, and I. Sirés, “ZnO/TiO2/Ag2Se nanostructures as photoelectrocatalysts for the degradation of oxytetracycline in water,” Electrochim Acta, vol. 331, Jan. 2020, doi: 10.1016/j.electacta.2019.135194.

H-New Developments in Battery Materials: Designing and Producing More Efficient Systems via Nanoscale Characterization

Controling the electronic properties of graphene nanoribbons towards energy applications.

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Graphene nanoribbons are quasi 1-dimensional materials consisting of strips of graphene with a defined width and edge structure. This geometry also defines their electronic and optical properties, which implies applications in various fields of nanotechnology. This work focuses on ultra-narrow graphene armchair nanoribbons (AGNRs) encapsulated inside single-walled carbon nanotubes (SWCNTs). These AGNRs belong to the N=3p+2 family, and are expected to have small but non-vanishing band gaps. It has been reported elsewhere that the Raman response of the precursor molecule that remains outside the tubes can be discerned from that of the inner GNR. Inspired by this possibility we have explored the influence of the encapsulation on the band gap of the GNR, which has never been measured using optical characterization techniques to the best of our knowledge. To better understand the properties of this one-dimensional material we have performed Fourier Transform Spectroscopy (FTS) on a broad energy range from 0.1 to 2.5 eV. We have found both a reduction and a shift of the E11 excitation of the SWCNTs as a result of the interactions between the ribbon and the host. A GNR band gap for encapsulated ribbons could be estimated to lie around 0.19 eV, smaller than for free standing GNRs as expected from DFT calculations. Optimizing the properties of such hybrid materials offers tremendous opportunities for applications in energy related applications.

Dynamic stability design for advanced solid-statebatteries

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Solid state battery is a mechanically more coupled device than the commercial liquid electrolyte Li-ion batteries. Mechanical constriction effect strongly modulates the electrochemical stability at all interfaces in a solid-state battery. This effect introduces some unique dynamical evolution of interface reactions upon battery cycling, understanding of which is challenging but also giving us an opportunity to design the battery performance beyond commercial Li-ion batteries. The talk discusses the design of "dynamic stability"; at various interfaces for a stable long cycling of Li metal anode solid state batteries, where Li dendrite penetration can be suppressed through this dynamic process at high current densities. Advanced characterization will be combined with simulation to elucidate mechanistic details.

Imaging Characterization Challenges and Solutions for Advanced Battery Development

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Lithium battery is an energy storage system with highly complex microstructures covering multiple length scales, namely from millimeters to nanometers. The performance of the battery is dependent on the battery microstructures. Therefore, accurately characterizing the battery microstructure at different length scales is critical for interpreting battery performance, which ultimately advances battery technology.

Imaging characterization via electron microscopy, including SEM, FIB-SEM, and TEM, has been recognized as an effective approach to studying battery microstructure. However, it is challenging to capture the materials at the full-length scale and also correlate each data set. In addition, some materials are beam-sensitive and air sensitive, making it challenging to observe the structure at its native state.

In this talk, I will discuss a couple of challenges in imaging characterization on battery materials and how to use a comprehensive approach to study the battery structure at multiple length scales in 2D and 3D. In addition, solutions such as cryogenic FIB and TEM, inert gas sample transfer, and dose management approach to observe the battery materials are their native state will also be discussed.

Understanding $LiNi_xCo_yMn_zO_2$ battery cathodes by advanced transmission electron microscopy

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Abstract

$LiNi_xCo_yMn_zO_2$ (NCM), a common cathode battery material, exhibits a specific capacity and operating voltage comparable to $LiCoO_2$, while being less toxic. It is reported that NCM is composed of a mixture of $LiMO_2$/ Li_2MnO_3 layers (where M=Ni, Co or Mn). $LiMO_2$ is associated with a trigonal (R3m) phase, whereas Li_2MnO_3 is associated with a monoclinic (C2/m) phase, which is responsible for storing and providing Li+ during Li-extraction of $LiMO_2$. Nevertheless, these layered materials exhibit a large degree of cation disorder, which disrupts the Li+ pathways and creates a continuous MO_2 layer, lowering the Li mobility and reducing the cycling performance. Yet, so far, these phenomena have been mostly studied in single-crystalline particles. However, at the industrial scale, the NCM produced is typically in the form of micrometer polycrystalline particles, where the primary particles possess a very complex microstructure, in particular an agglomeration of nanoparticles, porosity, as well as chemical and phase heterogeneity. In this regard, this work aims to fundamentally understand the changes in chemical distribution as a function of Ni content in polycrystalline NCM cathode materials. In particular, the chemical composition and structure of the NCM polycrystalline particles with Ni contents from 0.70 up to 0.90 wt. % were investigated using FIB-SEM, aberration-corrected STEM, EDS mapping and precession electron diffraction. Slice-and-view SEM/FIB analysis revealed a geode-like morphology, independently of the composition. Yet, as the Ni content increases, the particle porosity decreases. STEM-EDS revealed variations in chemical composition across a single particle, in particular Ni content. Despite precession electron diffraction results suggesting the presence of only one crystal structure, atomic-resolution STEM images revealed that variations in the composition may be related to the presence of orientation variants within a single crystalline grain.

Emerging strategies and technologies for TEM imagingof beam and air sensitive materials

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Designing less expensive, more efficient, and more reliable energy storage devices requiresa deeper understanding of the structural and chemical properties of the constituent materials. This area of research has been particularly challenging using traditional electron microscopy characterization methods, as many of these materials are highly sensitive to air and moisture, as well as the electron beam at standard dose levels.

Preserving these materials in a fully inert atmosphere throughout the sample preparation, imaging, and analytical stages requires improved integration and hardware interfacing between complementary techniques. Once in the TEM, precise measurement and control of the electron dose is critical to reproducible experimentation. This is achieved through new software innovations and improved source and detection hardware.

Large solid-angle EDX detectors are needed to map out sample chemistry in 3D without damaging the structure. Dose-efficient imaging techniques such as integrated differential phase contrast (iDPC) coupled with new single-electron sensitive detectors have been key advancements to resolving structure of delicate materials. Furthermore, working at cryogenic temperatures is often a requirement alongside these strategies to ensure sample integrity and validity of experimental results.

Nanoscale structural analysis of iron oxide crystallites using electron diffraction-based Pair Distribution Function (e-PDF) technique

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Magnetic materials of spinel iron oxide compounds such as magnetite and maghemite have recently received immense interest due to their potential applications in various fields. Studying the atomic structure of these nanomaterials is essential to understand their unique properties. Traditionally, the structure of the material is determined from the Bragg peaks in the diffraction patterns. As the size of the nanomaterial is reduced, the fine powder crystallites lack structural coherence, and the diffraction patterns show broad Bragg peaks along with a pronounced diffuse component. This renders the application of classical powder diffraction techniques to be challenging for structural characterization. To overcome this, considering both Bragg and diffuse scattering, different total scattering methods were developed, and Pair Distribution Function (PDF) is one of them. Due to theability to deliver a large elastic momentum transfer range (q_{max}), neutron and synchrotron sources are generally considered to be favorable options for PDF studies. However, with theadvent of superior detector systems and better control of parallel or tilted electron beams inaberration-corrected TEMs, it is now a common practice to obtain $q_{max}$of 30 Å^{-1}and performhigh real space resolution e-PDF studies. Given this, the large scattering cross section associated with electrons is generally subjected to multiple scattering effects. Previousworks show that the electron scattering related to dynamical effects can be limited by reducing the specimen thickness, but with the change in electron energy, its impact is notvery clear. In the present work, the multiple scattering effects in 4 - 5nm sized iron oxidecrystallites were explored, and quantitative data treatment of e-PDF profiles acquired atdifferent beam energies is illustrated.

Poly(o methoxyaniline) modified by the BiphasicCeramic System $Na_2Ti_3O_7$/ $Na_2Ti_6O_{13}$

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POMA is a conductive polymer similar to polyaniline and presents electrical conductivities in the range of 10^{-1}-10^{-4} S cm^{-1}[1]. On the other hand, sodium titanates are semiconductor ceramics reported as good alternatives for application as solid electrolytes, with electrical conductivity values of 10^{-6} S cm^{-1}[2]. We probed the structural modifications and electronic behavior of composites obtained from the mixture of a biphasic titanate ceramic system ($Na_2Ti_3O_7$/$Na_2Ti_6O_{13}$) with a poly(o-methoxyaniline, POMA) matrix. The composites were obtained by the addition of $Na_2Ti_3O_7$/$Na_2Ti_6O_{13}$ to a POMA matrix in mass concentrations from 0 to 35 %. The sodium titanates were synthed by means of sonochemistry, while the polymer was obtained by conventional chemical polymerization. The structural analysis carried by XRD, FTIR and UV-Vis indicated mostly the presence of POMA, though the titanates could be identified in the composites with higher concentration. The XRD difractograms showed narrow reflections that could be correlated to the $Na_2Ti_6O_{13}$ phase and hydrogen titanates, whose presence indicates a process of partial substitution of H^+ for Na^+ ions. While the thermal degradation of the polymeric chains starts near 150 °C, the thermogravimetric and diferential thermogravimetric (TG/dTG) analysis revealed that the presence of sodium titanates enhances the thermal stability of the composites. Finally, the addition of the biphasic ceramic to the composite increases the electrical resistivity drastically, from 10^2 Ω cm in the pure POMA to 10^4 Ω cm in the 35 % composite.

Acknowledgements:

UFAM, FAPEAM agency and INDT.

References:

[1] M.M. Biondo, L.M. de Oliveira, S.X. Lima, A. de S. Carolino, A.L.F. Rocha, J.P. da Silva, Y.L. Ruiz, V.M. Giacon, P.H. Campelo, E.A. Sanches. J. Mol. Struct. 1205 (2020).

[2] L. Aguilera, N. Fagundes, A.D. Melo, B. Bandeira, F.X. Nobre, J. Anglada-Rivera, J.P. Silva, J. Pérez de la Cruz, Y. Leyet. Ceram. Int. 46 (2020) 8706-8710.

Solid-state-biofuel cell for powering implantablemedical devices

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Fuel cells are electrochemical devices that use redox reactions to convert ionic currents into electricity. It is a clean and sustainable energy because it generally uses hydrogen in the protonation with oxygen gas, obtaining water and non-toxic gases as products [1]. This energy can power an airplane, truck, car, electric bicycle and also a scooter. Currently, there are some studies to integrate biofuel cells to the implantable medical devices (IMD), such as stents, pacemakers, cardio-defibrillators and robotic prostheses, but these researches are in an early stage [2]. In the state-of-the-art highlight, an artificial blood solid-state-biofuel cell to power an IMD have been developed. Polycaprolactone (PCL) was subjected to electrospinning to form a nanofiber membrane. A part of it was modified with metallic sodium as anode electrode and hydrogen storage. Other part, it was modified with 5 W laser semicondutor to obtain laser induced graphene (LIG) as cathode electrode and oxygen diffusion. Furthermore, a proton exchange membrane (PEM) by incorporating hydrogen protons inside the PCL/poly-oligopeptide blende have been obtained. The edges of the solid-state-biofuel cell (1 cm^2) were sealed by a thermal junction with PCL (60 to 70 ºC) and characterized on artificial blood samples for potential and electric current measurement.

References:

[1] I. Staffell, D. Scamman, A.V. Abad, P. Balcombe, P. E. Dodds, P. Ekins, N. Shah, K.R. Ward. The role of hydrogen and fuel cells in the global energy system Energy Environ. Sci. 12 (2019) 463-491.

[2] R. Ibrahim, N. Shaari, A.H.M. Aman. Bio-fuel cell for medical device energy system: A review, International Journal of Energy Research 15 (2021) 14245-14273.

Theoretical Study of the solid-state electrolyte $Li_3PS_4$and the interfaces formed with Li_2S and Li-anode

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In recent years, solid-state lithium batteries (ASSLBs) have been investigated for their various qualities, including safety as they are non-flammable materials. Among them, sulfur-based solid electrolytes, such as lithium thiophosphate, Li_3PS_4, have drawn attention due to their good ionic conductivity, (the β-structure has an ionic conductivity of about 3.0 $x10^{-2}$ Scm^{-1} at 573 K)[1]. However, one of the problems that arise when it comes to ASSLBs is thatof interfaces and their defects, in particular the Li dendrites, which can compromise the correct functioning of the battery and the Li-ion migration. In this sense, understanding how interfaces and their defects are formed, and how we can avoid them, is a fundamental point for the advancement in the development of commercial batteries. Therefore, this work aims at exploring, from a theoretical point of view, the formation, stability, and properties of the interfaces formed between the stable surface (110) of β-Li_3PS_4 solid electrolyte[2] with the Li-anode and Li_2S, a passivating material that should stabilize the Li_3PS_4/Li-anode interface. Furthermore, possible pathways for the migration of Li-ions across the interfaces were hypothed based on the electron density critical points obtained through topological analysis.

Acknowledgments:

This research was funded by European Union' s Horizon 2020 research and innovation program, project MODALIS2, grant agreement No. 875193.

References:

[1] Homma, K.; Yonemura, M.; et al., Solid State Ion., 53-58 (2011).

[2] Marana, N. L.; Sgroi, M. F.; et al., Nanomaterials, 2795 (2022).

Effect of $Na_2Ti_3O_7$/ $Na_2Ti_6O_{13}$ incorporation in the properties and surface of PVA matrix

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Membranes obtained by the addition of sodium titanates ($Na_2Ti_3O_7$/$Na_2Ti_6O_{13}$) in a polyvinyl alcohol (PVA) matrix of are reported for the first time. The synthesis of sodium titanates occurred through high-intensity ultrasonic irradiation and subsequent heat treatment. PVA is a polymer used in solid electrolyte membranes because of its non-toxicity, easy formation of thin films, good water solubility, high chemical resistance, high tensile strength and flexibility and good capacity for storing charges[1-2].On the other hand, sodium titanates are semiconductor ceramics reported for application as solid electrolytes, with electrical conductivity values of 10^{-6} S cm^{-1}[3].Then, the three types of composites were produced using the solvent casting method: a sample of PVA polymer, a second PVA composite with the addition of $Na_2Ti_3O_7$/$Na_2Ti_6O_{13}$without heat treatment (NTO-WTT), and a third sample with heat-treated $Na_2Ti_3O_7$/$Na_2Ti_6O_{13}$. The analysis by FTIR allowed the identification of functional groups of the PVA polymeric chain, and the presence of the two sodium titanates phases RAMAN were detected by RAMAN. AFM showed n increase in the mean surface roughness of the PVA (~ 1.9 nm) to the maximum value of ~ 40.1 nm for the NTO-WTT sample. Finally, the sample NTO-WTT showed the lowest value of electrical resistance (10^5 Ω) and relaxation time (3.16 x 10^{-5} s). Therefore, the presence of these ceramic particlesin the PVA matrix improved its semiconductive properties and potential to electronic applications.

Acknowledgements: UFAM, FAPEAM agency and INDT.

References:

[1] Moly PP, Jeena CB, Elsa PJ, Ambily KJ, Joy VT. Polym Bull. 2018; 75(10):4409-4428.

[2] Soliman TS, Zaki MF, Hessien MM, Elkalashy SI..Opt Mater (Amst). 2021.

[3] L. Aguilera, N. Fagundes, A.D. Melo, B. Bandeira, F.X. Nobre, J. Anglada-Rivera, J.P. Silva, J. Pérez de la Cruz, Y. Leyet. Ceram. Int. 46 (2020) 8706-8710.

Functionalization, characterization and application of carbon nanotubes/sodium lignosulfonate dispersions

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Carbon nanotubes have excellent physical, chemical, mechanical, and thermal properties that make then attractive for many applications. However, the low interaction with most ofthe solvents and the tendency to agglomeration, make them difficult to use [1]. Thus, thisstudy aimed to functionalize the carbon nanotube with lignosulfonate and evaluate thecolloidal stability of the system. Dispersions with distinct nanotube amounts were prepared, using lignosulfonate as dispersing agent, with the aid of an ultrasound bath.

The effect of dispersion was evaluated using X-Ray Diffraction (XRD), Infrared Absorption Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA), Raman spectroscopy and Zeta Potential. The functionalization was confirmed by means of FTIR, TG and SEM. The addition of lignosulfonate does not change the crystallinity patterns of the nanotube and did not promote the introduction of defects, which was confirmed in the XRD and Raman techniques, respectively, indicating a non-covalent functionalization. Carbon nanotubes dispersions prepared with lignosulfonate showed good colloidal stability during several months, and were applied in negative electrodes of lead-acid cells. Well-distributed carbon nanotubes were observed within the bulk electrode, enhancing the efficiency to accept charge, especially in partial state of charge, which was confirmed by electrical charge acceptance tests.

Acknowledgements:

UFPE; INCT-NANOVIDA; FACEPE for promoting the scholarship.

References:

[1] DANESHVAR, F. et al. Critical challenges and advances in the carbon nanotube-metal interface for next-generation electronics. Nanoscale Advances, v. 3, n. 4, p. 942-962 (2021)

Hydrothermal synthesis of alumina nanoparticles for electrode coatings of Lithium-ion batteries

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Despite their low weight and long service life, lithium-ion batteries (LIBs) have some limitations such as degradation and risk of fire in case of overheating [1]. Some nanomaterials for coating electrodes have become a solution to improve the performance of LIBs. Among them, alumina (Al_2O_3) has stood out due to the reduction of electrode degradation during charge and discharge cycles, in addition to improving heat dissipation [1]. Al_2O_3 can be synthesized by several techniques, but the hydrothermal synthesis route has been highlighted due to the efficient control of the size and morphology of nanoparticles [2]. The objective of this work is to synthesize nanostructured Al_2O_3 by the hydrothermal method, at temperatures as low as 175°C for 1 h, using precursors such as aluminum nitrate and urea. X-ray diffraction (XRD) results indicated the characteristic peaks of γ- Al_2O_3. From the results of Fourier transform infrared spectroscopy (FTIR), the formation of O-H and Al-O bonds, which are characteristic of γ- Al_2O_3, could be verified. Scanning electron microscopy with field emission gun (FEG-SEM) showed nanoparticles with well-defined plaque morphology and low agglomeration.

Acknowledgements:

To the Human Resources Program of the National Agency of Petroleum, Natural Gas and Biofuels - PRH-ANP, for the financial support supported with resources from the investmentof oil companies qualified in the RD&I Clause of ANP Resolution nº 50/2015.

References:

[1] T. Lee, W.-K. Kim, Y. Lee, M.-H. Ryou, Y.M. Lee, Effect of Al2O3 coatings prepared by RF sputtering on polyethylene separators for high-power lithium ion batteries, Macromol. Res. 22 (2014) 1190-1195. https://doi.org/10.1007/s13233-014-2163-1.

[2] S. Minaei, M. Abdollahifar, T. Shojaeimehr, D. Kumar, Micro/mesoporous quasi-zero-dimensional AlOOH and Al2O3 nanoparticles, Inorg. Nano-Metal Chem. 50 (2020) 170-177. https://doi.org/10.1080/24701556.2019.1703000.

Inert gas sample transfer workflow for lithium-based battery characterization

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One of the key methods for achieving the understanding of the battery materials structure has been through techniques such as DualBeam (FIBSEM) lamella preparation and subsequent TEM analysis. However, due to the air and moisture sensitivity of certain battery components, maintaining sample integrity for characterization has been a challenging task. This poster will present an approach for analyzing lithium-based battery materials in their native state using an inert gas sample transfer workflow.

INFLUENCE OF pH ON ELECTRICAL PROPERTIES OF SODIUM TITANATE CERAMICS*M.A. F Santos¹, J.H.L. Silva¹, Y. Leyet-Ruiz², D.V. Sampaio3R.S Silva¹¹ Federal University of Sergipe - Brazil²Federal University of Amazonas - Brazil³Federal University of Alagoas - Brazil

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The development of efficient batteries has been a major challenge for industry and scientists, since they must meet certain criteria, such as: high efficiency - providing a large amount of energy at a good cost-benefit; be safe and have clean energy from renewable sources. Therefore, the study of ionic conductors has been important because they meet these requirements and have a high ionic conductivity, due to the movement of atoms or ions at temperatures much lower than its melting point. In recent years, special attention has been paid to sodium titanate ceramics (NTO) due to its high ionic conduction at room temperature. In this work, the influence of pH on synthesis, conventional and laser sintering, and electrical characterization of $Na_2Ti_3O_7$ ceramics was done. $Na_2Ti_3O_7$ powders were synthed by the polymeric precursor method, changing the pH of the solution from 0 (as prepared) to 11, and calcined at 500 °C for 5h. The ceramics were conventionally sintered in an electric oven at 1040 °C for 2 h and laser-sintered, using a CO_2 laser as heating source. In both cases the samples produced from pH 0 showed higher relative density and homogeneous microstructure. All samples produced showed majority the $Na_2Ti_3O_7$ phase. The electrical conductivity of the sintered ceramics was approximately 10^{-4}S. cm^{-1}.

Structural and electrical properties of polymer electrolytes synthed by solution casting

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In recent decades, with the advent of new electronic devices and the growing demand for energy, researchers have sought new sources of energy storage[1]. Since then, lithium-ion batteries (LIBs) have gained particular attention. Because of this, different electrolytes have been studied. Polymeric solid electrolytes have considerable advantages, such as high flexibility and low cost, being more suitable for mass production [2], receiving special attention. This work presents the synthesis and characterization of polymeric electrolytes in the form of lithium-doped films varying the proportion of polymers using the solvent casting method. This method is effective for forming a uniform film. Poly(vinyl alcohol)-PVA and poly(vinylidene fluoride)-PVDF were the polymers used and N-Methyl-2-pyrrolidone solvent was used as dispersion medium. The results of Complex Impedance Spectroscopy revealed that the electrolyte with 75%PVA 25%PVDF 1% Li presented low resistance in the order of 102, indicating in this proportion the formation of an electrolyte with good electrical properties. The X-Ray Diffraction technique revealed that there was the formation of the PVA and PVDF phase and the cohesion between them. The characteristic peaks of PVDF are centered at $2\theta = 18.3°$, $2\theta = 19.9°$, $2\theta = 26.06°$ and of PVA at $2\theta = 19.6°$ and 22.3°. Infraredspectroscopy is in accordance with the literature and presents profiles following a patternwhen PVA is added to the structure, appearing vibrational bands with changes in intensityin others. Finally, this work reveals that electrolytes with adequate proportion, have potential to be used in energy storage devices.

Acknowledgements:

Fundação de Amparo à Pesquisa do Estado do Amazonas (FAPEAM), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and UFAM for the financial supportand infrastructure.

[1] X.Yinsi and L.Jun. Colloids and Surfaces A. vol.632, 127773(2022).

[2] C.Jiahao and Z.Hong. Power Sources. vol.548,232109(2022)

Synthesis and Characterization of graphene-doped PVA-PVDF-Li polymeric electrolytes

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Many of the studies on polymeric electrolytes have been focused on enhancing their properties, such as increasing ionic conductivity at room temperature [1]. One way to achieve high ionic conductivity is by mixing polymers. These mixtures gave way to a new form of processing, becoming commercially and technologically more important than the manufacture of homopolymers and copolymers that until then were valued [1-2]. For this reason, this work presents the synthesis and characterization of polymeric electrolytes doped with lithium and graphene by the solvent casting method. Poly(vinylidene fluoride)-PVDF and poly(vinyl alcohol)-PVA were the polymers used in the proportion 75%PVA 25%PVDF with 9% Li. The amount of graphene doped was 0.5%, 1%, 1.5% and 2%. Results showed that the electrolyte with 1.5% graphene showed the best response in terms of electrical resistance, in the order of 101, possibly because graphene contributes to the dispersion between PVA and PVDF and conductivity in the order of 2.5 x 10^{-4} S/cm X-Ray Diffraction revealed the phase formation of the materials and confirmed the presence of graphene. Finally, this work reveals that the electrolyte 75%PVA 25%PVDF 9% Li - 1.5% graphene can be used as an electrolyte for Li-ion batteries.

Acknowledgements:

Fundação de Amparo à Pesquisa do Estado do Amazonas (FAPEAM), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and UFAM for the financial supportand infrastructure.

References:

[1] C. Jiahao and Z. Hong. Power Sources. vol. 548, 232109 (2022)

[2] P. Tamilselvi and M. Hema. Physica B: Condensed Matter. vol. 437, 53-57 (2014)

SYNTHESIS AND CHARACTERIZATION OF PVA/PVDF MEMBRANES BLENDED WITH LiOH

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The study of foldable batteries without safety risks is a crucial task for the realization of portable and wearable electrical devices. Given these concerns, the development of solid-state batteries is one of the most promising routes to achieving this quest. In this work, a study was carried out with polymeric electrolytes composed of poly (vinylidene fluoride) PVDF and poly (vinyl alcohol) PVA with different proportions, prepared by the solvent casting method, adding 1% of lithium salt (LiOH). X-ray diffraction (XRD) revealed that increasing the percentage of PVA in relation to PVDF showed a greater non-crystalline nature of the electrolytes and the polymer-salt interactions were analyzed by infrared spectroscopy (FTIR) where the structural characteristics of the polymers used were identified through the spectrum and the changes in bands related to each material. The determination of electrical properties and dielectric behavior were carried out as a function of frequency by complex impedance spectroscopy (EIC), where it was verified that the resistance decreased in the order of $10^3 \Omega$ for the sample with 75% PVA, 25% PVdF and 1 % of LiOH as the most promising result for this research.

Acknowledgements:

LPMAT research group, INDT and FAPEAM.

References:

[1] S. Rajendran, M. Sivakumar, R. Subadevi, M. Nirmala. Physics B: Condensed Matter. vol. 348, page 73-78 (2004).

[2] F. Alloina, C. Iojoiu. Polymer Electrolytes. Page 129-175 (2010).

[3] P. Tamilselvi, M. Hema. Physica B: Condensed Matter. vol. 437, page 53-57 (2014).

Synthesis of lithium-doped sodium titanate by sonochemistry method

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Currently, there is a pressing need for alternative energy sources in the global energy matrix, as approximately 80% of the world''s energy consumption is derived from burning fossil fuels [1]. In this context, research in the field of solid-state batteries has been gaining momentum, with sodium titanate emerging as a promising option for ceramic electrolyte production. In this work, lithium-doped sodium titanate was synthed using a sonochemical technique. The titanium isopropoxide was diluted in isopropyl alcohol and mixed with other components on a magnetic stirrer until homogenized, and then subjected to sonication for 15 minutes at a power of 487.5 W (65%). The samples were subsequently dried in an oven for 24 hours at 120 °C. A heat treatment was carried out at 900 °C for one hour. XRD measurements were performed in the non-doped sample and in the sample doped with 1% lithium followed by analysis using Rietveld refinement. In the sample without doping, sodium trititanate ($Na_2Ti_3O_7$) predominated, constituting 84.94% of the composition, along with sodium hexatitanate ($Na_2Ti_6O_{13}$) at 15.06%. In contrast, the sample doped with 1% of Lishowed an increased occurrence of sodium trititanate ($Na_2Ti_3O_7$) at 94.89%, withhexatitanate constituting only 5.11% of the composition.

The synthesis of sodium trititanate through sonochemical means is thus an excellent approach, owing to the high level of purity observed in the resulting samples.

Acknowledgements:

UFAM, FAPEAM agency and INDT.

References:

[1] Orsetti, N. Gabriel. The transcendental role of lithium zirconates in the development of modern energy technologies. Ceramics International Journal, 2022.

I-Materials for energy conversion and storage

Advances in Materials for Solar Photoelectrochemical Production of Fuels

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Solar water splitting and carbon dioxide reduction are two leading strategies currently pursued for fuel production aiming at net zero emission. Photovoltaics assisted, thermochemical, photoelectrochemical (PEC) and biomass based processes are primarily explored for achieving this goal. Among these, only PEC water splitting and CO2 reduction involve direct conversion of sunlight to fuels under atmospheric conditions. The PEC technology has not yet been proven commercially viable mainly due to the challenges posed by semiconductors used as photocatalysts. Recently emerged low dimensional materials and heterostructures offer promising pathways for solving these problems. Among various architectures, films of tubular nanostructures vertically aligned on substrates have exceptional properties including their extremely high surface area, improved light absorption and availability of charge carriers in the vicinity of the surfaces/interfaces to carryout redox reactions. In this presentation, we discuss the results of our simulation studies on electromagnetic wave propagation through these nanoscale materials and experiments to develop and characterize these materials for PEC fuel production.

Electrochemical Energy Storage: The Crucial Role of Batteries and Supercapacitors in Modern Technology.

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In this presentation, we will discuss the disruptive impacts of metal-ion batteries and electrochemical capacitors on technology and future projections for these technologies, including the development of new materials such as nanostructured silicon (Si). We will also examine the performance of two ionic liquids (ILs), P2225FSI and BMPYRFSI, as electrolytes for Si/polyacrylonitrile (PAN) composite electrodes, which demonstrated promising results with a specific charge capacity of about 1000 mAh g−1 and a coulombic efficiency of nearly 100% after 1000 charge/discharge cycles. We propose LixMn1.8Ti0.2O4@N-doped graphene oxide (x ≤ 1) as a superior positive electrode material for lithium-ion batteries, which showed excellent Li storage performance in organic media due to the incorporation of a conductive carbonaceous matrix. We also studied the Li storage capacity using ionic liquids as electrolyte solvents and found the best response at 40 °C with a specific capacity of 117 mA h g-1 at an applied current density of 1 C. Finally, wewill discuss the benefits and challenges of using ILs as electrolytes in lithium-ion batteries, including their high-rate cycling capabilities and practical applications in extreme environments. ILs offer improved safety, high thermal stability, and low flammability, but challenges such as high viscosity, low conductivity, and obtaining pure and stable ILs must be addressed. Overall, our findings suggest that ILs show great promise for the development of high-performance lithium-ion batteries with enhanced safety and stability.

Interrogating hybrid perovskites from the nano- to the macroscale

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Hybrid organic-inorganic perovskites (HOIPs) are a promising class of material for solar cells. Nevertheless, their long-term stability must be established prior to commercialization. Edisonian, traditional trial-and-error methods for material screening, development, and stability testing are slow considering the vast hyper-parameter space entailing chemical composition and the potential influence of environmental stressors when acted in combination. To overcome this bottleneck towards the identification of stable HOIPs, we realize automated experimentation and machine learning (ML) to gain physical understanding of how these materials' behavior is affected by environmental stressors, suchas relative humidity and temperature. We apply ML models to analyze high throughput, insitu steady-state photoluminescence and predict the changes in CsyFA1−yPb(BrxI1−x)3 perovskites while exposed to relative humidity cycles. We compare linear regression, echo state network, and seasonal auto-regressive integrated moving average with eXogenous regressor algorithms and attain consistent accuracy of >90% for the latter, while following long-term changes for 50 hours. Our accurate time series predictions showcase the promise of ML to mimic non-linear response from a series of hybrid perovskite compositions. At the nanoscale, we implement environmental scanning probe microscopy methods to quantify local changes in the electrical response of these materials. Overall, the suite of characterization methods presented here can be extended to other perovskite families, including Pb-free options.

Novel hybrid perovskites from ab-initio simulations

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Hybrid perovskites have shown great potential for photovoltaics, but one of themain issues is that the most active compositions contain lead. For this reason,substantial research efforts are devoted to the substitution of leadwith other elements, also in combination with vacancies. Here, we follow a differentroute, and, supported by ab-initio simulations, we propose that it could be possibleto produce hybrid perovskites where lead is substituted bya small atomic cluster. Simulations predict that some of these compounds can be dynamically stable and can display interesting properties for solar-energy applications.

Radical polymer devices for data and energy storage,and solar cell applications

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Organic polyradicals in which each unit is a stable paramagnetic radical that can be reversibly oxidized or reduced, are unique alternatives semiconducting polymers with diamagnetic repeating units that exist only with one state, which are at the basis of most polymer organic electronic devices proposed to date, including bulk-heterojunction organic photovoltaics (OPVs) and field-effect transistors (FETs). While the electrical transport properties in semiconducting polymers can be described by the formation of solitons or polarons along the chain, the electrical conductivity of polyradicals stems the oxidation of a radical site forming a cation, which can oxidize a neighboring neutral site, moving the oxidation even further along the chain in a dynamic redox process known as Marcus electron transfer. This model has enabled the design of redox flow batteries from a large class of polynitroxide radical polymers with stable neutral and cationic states at each repeating unit. Despite recent claims of p-type doping, all such materials have so far fallen short of enabling any electronic devices, such as FETs in which gate modulation and differentiated p-type and n-type transport electrons and holes is necessary. In this talk, we will review our work on electrotunable radical polymers with ambipolar characteristics, in particular poly-(oxo)verdazyls in which each repeating unit may stably exist in the anionic, neutral and cationic configurations. We show that due to the possibility of reversible disproportionation and comproportionation along the polymer chain enables both ambipolar and p-type field effect transistors. Furthermore, the same systems can be utilized in single-layer flow batteries, resistive memory devices and hybrid solar cells.

Acknowledgements: The hard work of graduate students and postdocs from my group is gratefully acknowledged. Gilroy's group from Western's Chemistry Department is recognized for their synthetic chemistry contributions to this project

Undoped and doped vanadium dioxide : synthesis and applications

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Vanadium dioxide (VO_2) is a "smart" material that exhibits a first-order reversible semiconductor-to-metal transition (SMT) at a temperature of $T_{SMT} \approx 68$ °C. This SMT is associated with a structural transition from a low-temperature (semiconductor) monoclinic phase to a high-temperature (metallic) tetragonal phase and is characterized by drastic changes in VO_2 electrical resistivity and in both its infrared (IR) and terahertz (THz) reflectivity. Furthermore, the SMT properties of VO_2, namely the transition temperature, the sharpness of the transition, the amplitude of the properties modification and the related hysteresis width, can be efficiently tailored through doping with an appropriate concentration of donors and/or acceptors and through a proper control of crystallinity, morphology and stoichiometry of the VO_2 films. In this presentation, we will review our recent achievements on the synthesis, characterization and applications of undoped and doped VO_2 films.

Unravelling the Catalytic Property of Copper-Based Materials for High Electrochemical Conversion of Nitrate Ions to Ammonia

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NH_3 is an essential raw material in fertilizers, chemical industry, and emerging energy conversion processes. As a dense energy carrier, interest is due to its high amount of hydrogen, high energy density, and easy storage and transportation. The electrochemical reduction of NO_3^- (NO_3RR) is considered an promising technology to generate NH_3, both from an economic and environmental point of view, since: the electrical energy needed in the process can come from renewable sources, for operating in ambient temperature and pressure conditions, the absence of greenhouse gases, the electrochemical conversion of NO_3^- is more efficient in terms of yield rate and faradaic efficiency than N_2 electroreduction. In this talk, we will discuss the NH_3 formation under two different copper-based materials: Cu/Cu_2O electrodeposited composite and Cu_2O nanocubes. Quantification of ammonia produced in the potential range of −0.1 to −0.8 V vs. RHE showed that it is formed in the entire potential range with a maximum Faradaic Efficiency of 87 ± 3 % at −0.4 V and 94 ± 4 % at −0.30 V, for composites and nanocubes, respectively. In situ Raman and FTIR indicate that the reduction of the copper oxides takes place in less negative potentials than the one under which hydroxylamine intermediate is detected, which points out for the importance of oxygen vacancies as active sites for the reaction. In fact, the formation of oxygen vacancies after electrolysis was confirmed by XPS measurements. Microscopy techniques show a modification of the electrode leading to a smoother surface after the electrolysis, which corroborates that the catalytic effect is not related to surface roughening, but to the creation of oxygen vacancies under cathodic potentials. The molecular characterization by DEMS demonstrates that the main pathway to the formation of NH_3 comes from the decomposition of hydroxylamine enhanced by the oxygen vacancies.

2D diamond-like functionalized nanosheets forhydrogen evolution reaction

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Two-dimensional (2D) materials have been widely investigated since the discovery of graphene in 2004 and are excellent candidates for energy storage and catalytic conversion applications. The physical properties of 2D materials can vary considerably depending on the synthesis and functionalization methods, where the origin of all these different properties is still an open question. Accordingly, it is essential to investigate and understand the properties of functionalized 2D structures. In this context, we explored the structural, thermodynamic, dynamic, elastic and electronic properties of diamond-like graphene and diamond-like silicene nanosheets functionalized with distinct types of atoms [1, 2]. We predicted nanosheets with interesting physical properties and potential applications in several fields, including van der Waals heterostructures and thermoelectric devices. In particular, we studied and demonstrated the diamond-like silicene nanosheet functionalized with aluminum atoms is a promising candidate for alkali metal ion batteries, mainly for sodium and potassium ion batteries [3]. Finally, our current results have shown potential for using them as an efficient hydrogen evolution reaction.

Acknowledgements:

This investigation was partially supported by Brazilian agencies CNPq and CAPES.

References:

[1] Ipaves, B., Justo, J. F., and Assali, L. V. C., J. Phys. Chem. C 123, 23195 (2019).

[2] Ipaves, B., Justo, J. F., and Assali, L. V. C., Phys. Chem. Chem. Phys. 24, 8705 (2022).

[3] Ipaves, B., Justo, J. F., and Assali, L. V. C., Mol. Syst. Des. Eng. 8, 379 (2023).

2D-materials based nanocomposite thin films for electrodes in aqueous and transparent Na- or K-ion batteries

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This work presents the preparation of 2D-based thin films materials through on a novel and almost unlimited platform developed in our research group, the so-called liquid-liquid immiscible route (LLIR) [1], and their specific application as electrodes to transparent aqueous sodium- or potassium-ion batteries [2-5]. The LLIR was employed to prepare transparent bi- or tri-components thin films between 2D-materials (MoS_2 or graphene) and carbon nanotubes, copper oxide or Prussian blue analogues, with fine control on the thickness, transparecncy and homogeneity. The evident effect of the nanostructured materials on some superlative properties observed in those electrodes will be demonstrated and discussed based on the structure/properties relationship. Specific capacity as high as 1316 mA h $g^{-1}$for Na-ion aqueous batteries have been found for a MoS_2/Graphene/Cu_2O tri-component nanoarchitected film, at current density of 100 mA g^{-1}[5], which are among the best results presented in literature for this kind of application. Acknowledgments: CNPq, CAPES, INCT Nanocarbono; INCT NanoVida

References:

[1] Zarbin, A.J.G., Liquid-liquid interfaces: a unique and advantageous environment to both prepare and process thin films of complex materials, Mater. Horizons, 8, 409, 2021.
[2] Schmidt, A.; Ramos, M.K.; Pinto, C.S.; Prereira, A.F.; Souza, V.H.R.; Zarbin, A.J.G.; Electrode fabrication at liquid interfaces: towards transparency and flexibility. Electrochem. Comm., 134, 107183, 2022.
[3] Husmann, S.; Ramos, M.K.; Zarbin, A.J.G.; Transparent aqueous rechargeable sodium-ion battery. Electrochim. Acta, 422, 140548, 2022.
[4] Schmidt, A. Ramos, M.K.; Ferreira, C.M.; Braz, B.A.; Zarbin, A.J.G.; Molybdenum-based materials/carbon nanotubes nanocomposites prepared as thin and transparent films for aqueous K-ion batteries, Electrochim. Acta, 387, 138500, 2021.
[5] Ramos, M.K.; Zarbin, A.J.G.; to be submitted, 2023.

A novel naphthalenediimide substituted with carboxylate groups as a building block for advancedmaterials for the energy sector

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We introduce here a novel ligand, N,N'-bis(4-carboxyphenyl)-1,4,5,8-naphthalenediimide (CNDI), which is a suitable compound for the preparation of new materials for the energy sector. The compound was shown to be an excellent ligand for metal organic frameworks (MOFs) and $CsPbBr_3$ perovskites. The compound was synthed in a single step and high yields by using molten imidazole as the solvent. Reaction of CNDI with zinc nitrate or zirconium chloride generated the MOFs CNDI-Zn and CNDI-Zr. The MOFs were characterized by x-ray diffraction, nitrogen adsorption isotherms (BET method) and UV-visible spectroscopy. The presence of micropores was evidenced by BET measurements. The MOFs will be tested for the separation of CO_2 from other gases. CNDI is also an excellent ligand for $CsPbBr_3$ perovskite nanocrystals (NC). Addition of CNDI to a $CsPbBr_3$ NC solution in toluene resulted in strong binding of the imide to the NC surface, resulting in photoluminescence (PL) quenching, which can be attributed to electron transfer (ET) from the $CsPbBr_3$ NC to electron-acceptor CNDI. These results show that CNDI has a great potential for the construction of solar cells.

Acknowledgements: the authors thank FAPESP for the financial support (grant No 2020/15230-5)

A secondary source niobium oxide for batteries

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Batteries that can charge more rapidly and present higher capacity are wanted for electronic devices and electric vehicles. These requirements could be addressed by evolving electrodes that permit fast and safe storage. Niobium-based oxide anodes have shown larger capacity and higher rate capability than commercialized anodes [1]. Niobium oxide, Nb_2O_5, exists in different crystalline polymorphs, which present different electrochemical behavior [1]. In this work, Nb_2O_5hydrates were used as the starting materials to study the calcination temperature effect on the structural properties and electrochemical performance of these polymorphs. The Nb_2O_5hydrates were obtained by extracting Nb from fines that are considered a sub product in the manufacture of FeNb alloys. Before calcination, the Nb_2O_5hydrates were analyzed by TGA. After calcination, all the Nb_2O_5samples were deeply characterized by Raman, FTIR, XRD, SEM and TEM. Their electrochemical performances were tested in cyclic voltammetry measurements. The sample that showed the best structural and electrochemical properties was selected to be used as anode in a Li-ion battery. Therefore, this work not only reports a highly promising anode for fast charge batteries but also purposes the obtention of a niobium-containing product from a secondary source contributing to the improvement and development of urban mining and circular economy.

Acknowledgments:

This work was financially supported by CNPq (408563/2022-2). The authors would like to acknowledge the Center of Microscopy at UFMG (http://www.microscopia.ufmg.br) for providing the equipment and technical support for experiments involving electron microscopy. The support of PPGEM/UFMG laboratories is also acknowledged.

References:

[1] L.Tongtong et al., Energy Environ. Sci. 15, 254 (2022)

Chiral cobalt oxide nanostructures templated by D- andL-cysteinylhistidinylalanine for spin selectivity in electron transport

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In the water-splitting process, the oxygen evolution reaction constitutes a chokepoint that affects the overpotential. Because of spin constraints, triplet molecular oxygen formation competes with the singlet-mediated formation of hydrogen peroxide. A common strategy to favor spin polarized reactions is the modification of achiral materials with enantiomerically pure chiral ligands. Otherwise, chiral templates, such D and L amino acids and peptides can be used to act as templates for enantiomeric crystal growing of metallic and metal oxide nanoparticles. Recently, we used animal and plant ferritin to assist the synthesis of chiral Co_3O_4quantum dots (QDs) [1]. Natural proteins formed unically by L-α-amino acids can not template the formation of enantiomeric pairs of crystals. We synthesized the enantiomeric tripeptide D- and L-cysteinylhistinylalanine (D- and L-CHA) that efficiently assisted the production of enantiomeric chiromagnetic nanocrystals of Co_3O_4.The bandgap energies obtained for the cobalt oxide are 1.68 and 3.4 eV when D-CHA was the template and 2.17 and 3.48 eV when assisted by L-CHA with bandgap energy differences ($\Delta E = Eg1 - Eg2$) of 1.72 and 1.31 eV, respectively. The X-ray diffraction spectra showed evidenced peaks are indexed to (311), (400), (511) and (440) assigned to Co_3O_4facets. Magnetic circular dichroism evidenced the enantiomeric crystal growing templated by D- and L-CHA. The enantiomeric material was also characterized by high resolution transmission electron microscopy (HRTEM). The next step is the application in the spin-polarized electron transport.

Acknowledgements: FAPESP 2017/02317-2, 2019/01425-1, and CNPq 313111/2021-9.

[1] Julia D. Bronzato1, Aryane Tofanello, Martha Oliveira, Jefferson Bettini, Adrianne M. M. Brito, Alexandre J. C. Lanfredi, Silgia A. Costa, Otaciro R. Nascimento and Iseli L. Nantes-Cardoso. Virucidal, photocatalytic and chiromagnetic cobalt oxide quantum dots. Applied Surface Science, 576, 151847, 2022.

Flash Sinter-Crystallization of $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ for all solid-state batteries

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The development of all-solid-state batteries (ASSBs) is a promising technology for replacing the current liquid electrolyte batteries due to their higher energy density, improved safety, and longer lifespan. However, the commercialization of ASSBs is hindered by the low ionic conductivity of solid-state electrolytes and poor electrical contact between the electrolyte and the electrodes. In this study, we explored the use of ***Flash Sinter-Crystallization***(FSC) to accelerate the crystallization process of lithium aluminum titanium phosphate (LATP), a promising solid-state electrolyte. We melted a stoichiometric mix of precursor oxides to obtain a glass precursor of Li_2O - Al_2O_3- TiO_2- $P_2O_5$and processed it into pellets. The pellets were subjected to FSC at low furnace temperature (470 °C) using various current densities (from 10 to 130 mA mm^{-2}) and with an electric field of 200 V cm^{-1}, and the resulting samples were analyzed by Differential Scanning Calorimetry (DSC), ex-situ X-ray diffraction (XRD), and Electrochemical Impedance Spectroscopy (EIS). Our results showed that FSC can enhance the ionic conductivity of LATP by promoting its fast processing. The best results were achieved when a condition of 50 mA $mm^{-2}$was used, with a total ionic conductivity at room temperature of 0.3 mS cm^{-1}(Ea = 0.31 eV) and a bulk conductivity of 1.4 mS cm^{-1}. Furthermore, the FSC process is significantly faster than conventional sinter-crystallization, which typically requires 950 - 1100 °C and 2 hours. In our case, the sinter-crystallization was completed at 470 °C within 300 seconds. Our study demonstrates the effectiveness ofFSC as a fast and efficient technique for the processing of solid-state electrolytes, opening up possibilities for cost-effective solid-state battery production and co-sintering of the cathode with the solid electrolyte.

High capacitance and expanded voltage window of MXene/Nanodiamonds supercapacitors in $AlCl_3$ electrolyte

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MXenes have demonstrated excellent performance as constituent materials for supercapacitor electrodes due to their 2D morphology, high electrical conductivity, and large redox-active surface [1]. One of the most outstanding achievements is the capacitance (C) obtained with 2D $Ti_3C_2T_x$ in H_2SO_4 electrolyte, which is related to the surface redox process caused by the intercalation of H^+ between $Ti_3C_2T_x$ layers. The intercalated protons react with the O terminations of MXene and form OH groups, thus changing the Ti oxidation state, which results in the pseudocapacitive behavior [2]. However, a narrow potential window of highly acidic electrolytes (under 1 V) is a limiting feature for many applications. Wider voltage windows are crucial for obtaining supercapacitors with superior energy densities (E), since it is proportional to the square of the voltage (V), according to the formula for a double-layer capacitor: $E = \frac{1}{2}CV^2$ [3]. In this scenario, materials and electrolytes that produce high capacitance and large potential windows are highly sought after. In this work, we used nanodiamonds to pillar the $Ti_3C_2T_x$ structure and obtain superior supercapacitor performance in $AlCl_3$ electrolytes. The superior performance relies on the combination of electrolyte formulation and electrode structure design. The pillaring effect prevents the restacking of MXene layers, reducing diffusion limitations and resulting in a high-rate performance with a capacitance of 235 F/g (561 F/cm^3). The use of 3 M $AlCl_3$ provided protons that contributed to the capacitance obtained and allowed an expansion of the potential window to 1.2 V, due to the lowered activity of water in the electrolyte.

Acknowledgments:
The authors are thankful for the financial support from FAPESP, CNPq, and MCTI-SisNano.

References:
[1] Anasori, B. et. al. Nat Rev Mater 2017, 2 (2), 16098
[2] Zhan, C. et. al. J Phys Chem Lett 2018, 9 (6), 1223-1228
[3] Wang, G. et. al. Chem Soc Rev 2012, 41 (2), 797-828.

Hydrogen storage properties of the high-entropy alloys derived from the $Ti_xZr_{1-x}CrMnFeNi$ system

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This study investigated the crystal structure and hydrogen storage properties of the high entropy alloy derivated from the $Ti_xZr_{1-x}CrMnFeNi$ system. The alloys, which had an AB2-type configuration (A: elements forming hydride, B: elements non-forming hydride), were selected by the CALPHAD method [1]. The selected alloys with different at.% of Zr and Ti were evaluated with respect to their microstructure differences and their possible effect on the hydrogen storage properties. The arc-melted Ti-rich alloys showed the presence of BCC and C14 Laves phase in different fractions. On the other hand, the Zr-rich alloys showed the C14 Laves phase, along with a secondary intermetallic NiZr phase. Hydrogen storage properties investigated through pressure-composition-temperature (PCT) absorption and desorption isotherms revealed that the Zr-rich alloy could absorb 1.8 wt% (H/M = 1.2) of hydrogen with fast kinetics at room temperature after a simple heat activation procedure. In contrast, Ti-rich alloys absorbed only 0.2 wt% (H/M = 0.1) with slow kinetics due to their poor activation ability and the possible formation of a titanium oxide layer. The Zr-rich alloys were able to reversibly absorb and fully desorb 1.6 wt% (H/M = 1) of hydrogen. Furthermore, Zr-rich alloys exhibited a very low hydrogen equilibrium pressure in the PCI isotherms. After full hydrogenation at room temperature, the initial metallic C14 Laves phases were converted into their respective Laves phase hydrides in both cases. Under cycling, the fractions of the secondary BCC, NiZr, and C14 Laves phase changed due to the lattice distortion to accommodate the hydrogen atoms. The microstructural analysis, before and after cycling, showed a very well-homogeneous microstructure and good distribution of elements for both Ti and Zr-rich alloys.

Acknowledgments: CAPES (001), FAPESP (2022/01351-0)

References: [1] D.B Miracle; O.N Senkov. A critical review of high entropy alloys and related concepts. Acta Materialia. (2016)

Laser-induced graphene-cobalt oxide composite electrodes directly produced on polyimide tape: towards the efficient and cost-effective green hydrogenproduction

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The Anthropocene is marked by the unmistakable trace of human actions in the terrestial environment. Thus, the transition to clean energy alternatives are essential for sustainable human development and the stability of the planet, and green hydrogen is a promising solution to address these challenges [1]. This study aims to develop materials for electrocatalysis of oxygen evolution reaction (OER) using the in situ CO_2laser synthesis. This technique has advantages front conventional film deposition, such as low energy consumption, scalability, and really low time scale, making it possible to produce materials that by other methods take hours, in fractions of seconds [2]. The graphene-based electrode (LIG) was obtained from the laser ablation of polyimide films, and by the same technique, controlling the growth of nanostructured or compact films of cobalt oxide on the LIG' s surface to obtain the LIG-CoO_xmaterial. Different experimental conditions were tested, such as laser power, deposition technique, precursors solutions and concentrations. The electrodes materials were characterized by Raman spectroscopy, X-ray diffractometry, Energy-dispersive X-ray spectroscopy (EDS) and the morphology was analyzed by scanning electron microscopy (SEM). The electrochemical characterizations of bare LIG electrodes in $K_4[Fe(CN)_6]$ 1 mmol.L^{-1}shows peaks separations comparable to commercially available rough carbon electrodes. The OER tests in KOH 1 mol.$L^{-1}$of the LIG-CoO_xreached the threshold current densities of 10 mA.$cm^{-2}$with overpotentials up to 600 mV lower than the bare LIG electrodes in optimal conditions. The oxide layer of the composite electrode also provided great stability over OER, overcoming the problems of swelling, peeling-off and irreversible oxidation of the graphene layers described in the literature [2].

Acknowledgments: FACEPE and CNPq

References: [1] Sun, X., Xu, K. 8, 657 (2018). [2] Lopes, D. V., Santos, N. 11, 4158 (2023)

Microwave-assisted synthesis of metal semiconductors heterojunctions with photocatalytic applications

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Using solar light in photo-induced processes is attractive since it is a clean and practically limitless energy source. Therefore, research on developing new photocatalysts capable of using this energy has recently been growing. Metal semiconductors have excelled in this field. However, creating materials that utilize the visible radiation component is a contemporary challenge. For this purpose, we discuss the heterojunctions based on titanate nanotubes/carbon nitride [1], bismuth vanadate/tungsten oxide [2], and bismuth niobate/silver nanoparticles [3]. These catalysts were synthesized through microwave-assisted routes, an emergent technique that quickly obtains the desired products with high selectivity, high yields, and low energetic consumption. Thus, several methodological studies can be carried out quickly, allowing the investigation of the ideal synthetic conditions to obtain nanomaterials with controlled morphology and structure. As a result, the establishment of new microwave-assisted synthesis protocols was achieved. These heterojunctions were characterized through several techniques and applied to the photodegradation of organic species and photoelectrochemical generation of H2. A correct band alignment between the heterojunction components is required for these applications. Typically, the photogenerated electrons must flow from the external semiconductor to the host semiconductor; the photogenerated holes must flow from the host to the external semiconductor. This charge separation increases the electrons lifetime by reducing the recombination rates.

Acknowledgments: FAPESP, CNPq, CAPES, UFABC.

References:[1] B. Rodrigues, et.al. J Nanostructure Chem, 10, 363 (2020).
[2] C. Claudino, et al. Mater Res Bull, 125, 110783 (2020)
[3] S. Oliveira, M. Kuznetsova, et al. Ceram Int (2022)

$Nb_2O_5.nH_2O$ decorated with Platinum Nanoparticles (PtNPs): An Efficient Catalyst for Sodium Borohydride-Mediated Hydrogen Evolution Reaction

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Climate change-related disasters have occurred worldwide due to the exhaustive use of fossil fuels. One promising fuel alternative is hydrogen (H_2). While $H_2$is considered a clean fuel, it presents several challenges due to its flammability and low energy density. Inorganic hydrides such as $NaBH_4$can be used as storage systems, but hydrolysis reactions needed to release $H_2$are slow and require catalysts [1]. Niobium (Nb) is a transition metal with a wide range of applications, and most of the reserves are in Brazil (98%), which also accounts for most of the world's production (93%) [2]. Investigations on Nb-catalyzed hydrogen production have used Nb in the form of complexes, nanoparticles and combined with other metals [3]. In this study, we synthesized metal nanocatalysts supported on $Nb_2O_5.nH_2O$ to accelerate the hydrolysis of $NaBH_4$. We reduced a metal precursor (Au, Pd, Ni or Pt) onto $Nb_2O_5.nH_2O$ using a $NaBH_4$solution. The freshly prepared material was then added to a kitasato flask coupled to a burette filled with water. The container was closed, the $NaBH_4$solution was added, and the system was kept under stirring. The amount of H_2released was determined by measuring the water displacement in the burette, and the reaction was monitored until it reached completion. The average rates for H_2production at room temperature were 0.008, 0.051, 0.103, and 1.03 mL H_2 $s^{-1}$for Au (5 mL of H_2), Pd (18 mL of H_2), Ni (37 mL of H_2), and Pt (38 mL of H_2), respectively. We found that PtNPs on $Nb_2O_5.nH_2O$ had a significant catalytic effect and are a promising candidate for further investigations for fuel cell applications.

Acknowledgements: FAPEMIG (RED-00144-22), CNPq (405828/2022-5) and CAPES.

References

[1] S. S. Muir , X. Yao. Int. J. of Hydrog. Energy 36, 5983 (2011).
[2] P. M. Monte et al., Rev.Virtual de Quim. 13, 1391 (2021).
[3] A. Islam et al., J. Clean. Prod. 318, 128439 (2021).

$PtSe_2$ Nanoparticles and Their Structural Defects Towards Hydrogen Evolution Reaction

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Transition metal dichalcogenides (TMD) materials can generate 2D nanostructures due to their weak van der Waals interaction between monolayers. The $PtSe_2$ synthesis and characterization have not shown as much progress as other TMD like MoX_2 or WX_2 (X=S or Se) possibly due to the difficulty to obtain undamaged monolayers. However, the defects created can also be explored for several applications making $PtSe_2$ an interesting material to be explored. The hydrogen evolution reaction (HER) is one application of interest and catalyst engineering using defects could improve the process' s effectiveness. Here, a new synthesis protocol for $PtSe_2$ nanoparticles through the selenization of previously synthesized Pt nanoparticles was explored. The selenization process was performed in a closed reactor using an excess of Se at high temperatures (400 °C). The excess of Se was removed by solubilization in trioctylphosphine. A hexane solution of butyl-lithium and an ultrasonic bath was used to induce defects. $PtSe_2$ was characterized by HRTEM, EDS chemical analysis, and Pair Distribution Function from Electron Diffraction (ePDF) technique. HRTEM images revealed that after the lithium treatment only the short-range ordering (SRO) was kept showing a high concentration of defects. EDS indicated that Se vacancies are induced in the structure ($PtSe_x$), which was corroborated by XPS results. The new $PtSe_x$ structure presented a Raman Spectroscopy shift on both E_g and A_{1g} peaks and the E_g/A_{1g} intensities ratio was also affected. A new peak appeared in the spectrum assigned to the LA band (Longitudinal Acoustic Mode) from defects. The catalytic experiments showed an overpotential of 330 mV at 10 mA cm^{-2} for the $PtSe_2/PtSe_x$ nanoparticles compared to 90 mV for Pt Nanoparticles. We hope that future $PtSe_x$ defect engineering and catalyst optimization in the electrode could improve the HER performance.

Acknowledgements:
FAPESP (2021/03321-9) and FINEP (MQSM).

Reactive flash sintering for producing Li-ionconductors

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Flash sintering (FS) [1] consists of applying an electric field while heating a green ceramic sample. This technique has been used to sinter several materials at low temperatures in a matter of seconds, being cost-effective. Recently, FS has been extended to enable the simultaneous synthesis and sintering of ceramics in a derived method called reactive flash sintering (RFS) [2]. Herein, this approach is used for producing two Li-ion conductors: $Li_{0.5}La_{0.5}TiO_3$(LLTO) and $Li_{6.25}Al_{0.25}La_3Zr_2O_{12}$(Al-LLZO). Highly dense single-phase ceramics are obtained in a few seconds, with bulk ionic conductivity of 0.18 and 0.5 mS cm^{-1} for LLTO and Al-LLZO, respectively. Additionally, in-situ synchrotron X-ray diffraction is used to investigate the phase evolution. We show that RFS not only accelerates synthesis and sintering rates but also affects the reaction pathway of these complex oxides. Therefore, the enormous potential of this technique for producing materials with volatile compounds and for stabilizing phases that are not thermodynamically favored upon conventional heating is highlighted.

Acknowledgments:

FAPESP [grant No. 2018/19370-6] and CNPq [grant No. 403978/2021-1].

References:

[1] M. Cologna, B. Rashkova, R. Raj, J. Am. Ceram. Soc. 93, 3556 (2010).

[2] B. Yoon, V. Avila, I.R. Lavagnini, J.V. Campos, L.M. Jesus, Adv. Eng. Mater. 25, 2200731 (2023).

Reactive transition metal-oxygen species: a gas-phase study based on X-ray absorption spectroscopy

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Discovering compounds that present transition metals with unusual oxidation states or reactive oxygen species (superoxide, peroxide and oxygen-centered radical) is of great scientific and technological interests, as they have key applications as oxidizing agents, catalysts, or reaction intermediates, and can improve the performance of materials for energy conversion and storage.[1]

Here, we use X-ray absorption spectroscopy (XAS) at the oxygen K and metal L_3, M_3or N_3edges of gas-phase $[MO_n]^+$systems (M = transition metal, n= integer) to identify the spectroscopic signatures of oxygen ligands and assign the oxidation state of the metal.[2] Our ion trap is installed at the undulator beamline UE52-PGM at the Berlin synchrotron radiation facility BESSY II operated by the Helmholtz-Zentrum Berlin.[3]

Reactive species, such as oxygen-centered radicals and species containing high-valent transition metals, are analysed in stable conditions in the ground state inside the cryogenic ion trap. This method is here demonstrated to be an important tool to identify the character of oxygen ligands, offering direct access to element specific electronic structures.

Acknowledgments:

Ingo Krossing, German Federal Ministry of Education and Research (Grant No. BMBF-05K16VF1), DFG RTG 2717, ERC Project HighPotOx (818862), High-Performance Computing Center at ZEDAT (Freie Universität Berlin).

References:

[1] Y. X. Zhao, et al., Phys. Chem. Chem. Phys. 13, 1925-1938 (2011)

[2] M. da S. Santos, et al., Angew. Chem. Int. Ed., e202207688 (2022)

[3] K. Hirsch, et al., J. Phys. B: At., Mol. Opt. Phys. 42, 154029 (2009)

Simulating growth of nanostructured materials torelate their microscopic dynamics with their morphologies

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This presentation summarizes recent works that describe the morphology of growing materials using simulations of kinetic models which were properly designed for each deposition technique. First, we study island growth, coalescence, and initial formation of continuous heteroepitaxial films with a vapor deposition model [1]. A framework is proposed to relate the evolution of the surface roughness and of height correlations with step-edge energy barriers and atom/molecule diffusivities on the substrates. When those barriers are negligible, the roughness shows a maximum as the islands coalesce, as observed in hot-wall CdTe deposition on polyimide substrates and in thermally evaporated perovskite films. Next, using models of electrodeposition with adsorbate diffusion, we show initial compact film formation followed by instability development and dendrite growth [2]. The application to silver electrodeposition on nanoparticulated gold substrates explains the transition from microparticles with (111) facets to pine-tree shaped dendrites which preferentially grow from the corners and edges of those particles [3]. The approach shows that estimates of adatom diffusion coefficients in the electrodeposited structures can be obtained from their sizes and the applied currents. Finally, using a model of sputter deposition, we recently obtained morphologies similar to those of Si films with Ti nanoparticle scaffolds, which were developed for batteries. The multi-institutional Brazilian team currently involved with these modeling approaches is prone to begin other collaborations to describe the growth of novel materials.

Acknowledgements:
The author''s research is supported by FAPERJ, CNPq, and CAPES.

References:
[1] T.B.T. To, R. Almeida, S.O. Ferreira, F.D.A.A. Reis, Appl. Surf. Sci. 560, 149946 (2021).
[2] I.S.S. Carrasco, S.G. Alves, F.D.A.A. Reis, PCCP 24, 27856 (2022).
[3] S. Dokhan, D. di Caprio, A. Taleb, F.D.A.A. Reis, ACS Appl. Mater. Interfaces 14, 49362 (2022).

Techniques to improve the electrochemical stable potential window of EDLCs while keeping the capacitance and series resistance at high rates of discharge.

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This study aims to improve the electrochemical stable potential window (ESPW) of electrochemical double-layer capacitors (EDLCs) while maintaining high capacitance and low series resistance at high discharge rates. The first approach involves the use of inexpensive and non-corrosive lithium sulfate electrolytes, which allow for the application of diverse current collectors and simplify the capacitor assembly process. Electrochemical investigations demonstrate the feasibility of using 1 mol L-1 Li2SO4 aqueous solution for efficient capacitor operation in a wider voltage range, up to 2.0 V. The second approach involves a simple KOH treatment at 800°C of commercial activated carbon, resulting in highly pure carbons with effective surface areas in the range of 1300 m2 g−1 andgravimetric capacitances up to two times that of raw carbon. Re-activated carbons exhibitstable performance for high charge/discharge load in aqueous media at high current densities, achieving capacitance values of 90 F g−1 at 20 A g-1. This research presents apromising technique for improving the performance of EDLCs with potential applications invarious energy storage systems.

Acknowledgments:

Dr. Jian Liu (University of British Columbia-Canada); Dr. Noel Díez (Instituto Nacional del Carbón, Spain); Dr JR Miller (JME, Inc., United States); Dr. Jim Benson (Birla Carbon, USA)

References:

[1] K. Fic, G. Lota, M. Meller, and E. Frackowiak, Energy Env. Sci, vol. 5, no. 2, pp. 5842-5850, 2012.

[2] G. Lota, T. A. Centeno, E. Frackowiak, and F. Stoeckli, Electrochimica Acta, vol. 53, no. 5, pp. 2210-2216, 2008.

[3] M. Sevilla, G. A. Ferrero, and A. B. Fuertes, Carbon, vol. 114, pp. 50-58, 2017.

Unveiling the lithium-sulfur batteries: insights from a multidimensional in-situ and operando cell

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There is increasing interest in positive conversion electrodes for Li-S batteries. These batteries offer high specific capacity, low cost, and abundant sulfur. However, they face challenges such as the shuttle effect, where dissolved polysulfides migrate and degrade performance. One solution is to confine sulfur in porous structures [1]. Understanding Li-S battery mechanisms and degradation is vital. X-ray imaging, like CT, reveals morphological changes. In situ and operando experiments combine electrochemical and imaging methods, providing crucial insights for Li-S challenges [2]. In this research, we utilized a mechanochemical synthesis to create a Porous Organic Polymer (MTP-1) that acts as a host material for sulfur, serving as a positive electrode. We then employed microCT or Synchrotron Radiography and EIS simultaneously during cell operation to investigate the structural changes in sulfur and the internal resistance of two different cells: 1) Carbon Black/Sulfur (CB/S8) as the standard, and 2) MTP-1/Sulfur (MTP-1/S8). Analysis of electrochemical data and radiography images revealed superior sulfur handling by MTP-1/S8 in comparison to CB/S8 cells. MTP-1/S8 showed smaller sulfur crystals and favored the growth of alfa-sulfur (Synchrotron), which is more stable. Impedance spectroscopy showed MTP-1/S8 had lower resistance and better sulfur confinement than CB/S8. An interfacial barrier at negative electrode was observed in CB/S8 but not in MTP-1/S8. These findings demonstrate MTP-1's potential as a sulfur host material, improving capacity retention and electrochemical performance in Li-S batteries. Its ability to effectively accommodate sulfur and mitigate the shuttle effect leads to higher specific capacities and improved charge storage. Acknowledgements: FAPESP (2019/09341-1, 2021/11814-5), IQ-USP, HZB. References: [1] Souza, B.L. and Chauque S., JEC 896., 115459(2021), [2] Risse, S., PCCP 18., 10630(2016).

3D printed systems for laboratory scale-up studies of water splitting

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Green hydrogen production is essential for more sustainable routes to various sectors of the economy, but requires large investments and scale-up strategies [1]. To reduce production costs, the development of more efficient catalyst is critical, but the performance of laboratory tests does not reflect the same response in larger scale tests. For this reason, it is necessary to perform experiments on energy conversion devices, such as electrolyzer devices [2]. In this regard, the additive manufacturing technique has been widely used to produce electrodes and devices for rapid and inexpensive prototyping [3]. In this work, commercial conductive filament electrodes from Proto-Pasta© were printed by FDM technique and electrochemically characterized for HER and for OER. Then, a modular electrolyzer device was printed with ABS filament and the Proto-Pasta electrodes wirh geometric area of 2.5 cm2 were evaluated to understand the capabilities and limitations of the printed system in H2 production. The polarization curve was obtained at 20 °C with 1.0M KOH electrolyte for current density of 10 mA cm-2 (Ecell = 5.0 V vs. RHE). Mathematical models of electrolizer devices were used to evaluate the activation and ohmic overpotentials constributions. Finally, the application of the printed system for catalyst perfomance studies is proposed.

3D printing-assisted sacrificially templated piezoelectric scaffolds for cell regeneration

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The use of smart materials in tissue engineering has gained attention due to their ability to provide additional functionality and control over cellular fate. Piezoelectric materials offer a paradigm shift by delivering electrical signals to cells through mechanoelectric transduction, aiding tissue regeneration. In this work, we developed piezoelectric scaffolds with two controlled porosity based on gyroid surfaces using polydimethylsiloxane (PDMS) and sodium niobate ($NaNbO_3$). The gyroid-type negative mold was obtained by fused filament fabrication, resulting in a 5-mm-high x 10-mm-dia cylindrical scaffold. The $NaNbO_3$was hydrothermally synthed and incorporated at 30 wt% into PDMS. X-ray diffractograms confirmed the conversion of the initial crystalline structure of $NaNbO_3$to thepiezoelectric orthorhombic perovskite phase. Cubic morphologies with an average of 1.2 μmwere observed after $NaNbO_3$synthesis. Good distribution and dispersion of $NaNbO_3$within the PDMS matrix were confirmed by SEM-FEG and μCT, with the latter also confirming thetrue porosity of the scaffolds: 18 and 63%. Cyclic compression assays showed that increasedporosity led to decreased energy absorption, resulting in decreased damping capacity. After100 cycles, the scaffold bearing the lowest porosity (18%) exhibited the lowest loss of damping capacity (10%), while the 63%-porous showed a reduction of up to 24%. Young''smodulus increased from the absolute value of 0.01 to 0.07 MPa with decreasing porosity,respectively. To summarize, piezoelectric scaffolds were fabricated using PDMS and $NaNbO_3$via 3D printing-assisted sacrificial templating. Porosity was correlated with damping in mechanical compression, providing an important approach to meet the mechanical requirements of scaffolding and the potential to correlate with the electrical output of thepiezoelectric device.

Acknowledgments:
CAPES, CNPq (grant no. 304753/2022-0), FINEP (MATENERGIA, grant no. 01.22.0177.00).

Ab-initio calculations of ion occupancy at interstitials sites in the Prussian Blue framework

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Prussian Blue (PB) and its analogous are fascinating materials, rich in new magneto-optical and electrochemical phenomena, caused mainly by the mixed valence of their iron atoms. The importance of PB also comes from its large interstitial sites in the cubic crystalline structure, which can be empty or filled by alkali metals through redox reactions (intercalation). The great appeal of PB electrodes is the possibility to reach high charge capacities, near to Lithium (Li) batteries, by using rather environment-friendly elements such as Sodium (Na) or Potassium (K), in water-based electrolytes. In this study, Density Functional Theory (DFT) was applied to evaluate the crystalline structure and cell distortions of PB and Prussian White (PW) framework, regarding the presence of Li, Na, K, and Rb atoms. The calculations used the VASP package, plane waves basis and periodic boundary conditions. The exchange-correlation functional was a Perdew-Burke-Ernzerhof (PBE) type, using the Projector-Augmented-Wave (PAW) method to treat the core electrons. A single cell of 56 atoms was calculated and the K space grid was set to have about 64 points. We performed ionic relaxation for PB and PW cubic phase with ion intercalation in three interstitial sites: 8c, 24d, and 32f' site (Wyckoff notation). We observed that independently of the cation on the 8c site, the most stable conformation is a cubic phase, for both PB and PW. The same trend is observed for the 32f' site, where the stabler phase shows rhombohedral symmetry, independently of the cation on the interstitial sites and compound. However, the final phase for cations in the 24d site appears to be compound dependent: for PB, the phase was monoclinic, whereas for PW the phase is always tetragonal, regardless of the ion type. These results could help to understand the presence of crystalline phase transition in charge/discharge of PB and PW as well as the large dispersion of results of charge capacity found in the literature.

Activated biochar as low-cost catalyst for catalytic intermediate co-pyrolysis of waste plastic and biomass

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The state of Espírito Santo, one of the top producers of coconuts in Brazil, generates a lot of waste from processing and consumption [1]. The use of green coconut pericarp as feedstock in pyrolysis processes was investigated, focusing on turning these residues into valuable products such as bio-oil and catalysts. Waste polystyrene [2] and biochar-based catalysts [3] were employed on catalytic intermediate co-pyrolysis to examine their influence on both the yield and composition of the oil produced. Biochar from intermediate pyrolysis was directly employed as a catalyst, but steam-activated biochars with different activation times from single-step activation were also studied as low-cost catalysts. The properties of the catalysts were characterized by N2 physisorption, SEM, SEM-EDS, XRD, and FTIR analyses. From these techniques, AAEM (particularly K) and O-containing functional groups were identified on both biochar and activated biochars, but the activated ones presented a higher catalytic performance due to their larger surface area (>600m2/g). The results of co-pyrolysis assays showed that biomass and plastic produced a higher yield of oil and that the composition of the oil shifted away from oxygenated compounds [2]. Catalytic co-pyrolysis oils produced more monoaromatics when activated biochar was used as a catalyst, compared to other catalysts reported in the literature [2,3]. This effect was attributed to a synergetic effect between waste polystyrene and activated biochar, which resulted in an oil organic fraction containing up to 60% of xylene in its composition and presenting a HHV of 34.11MJ/kg.

Acknowledgments:
CAPES for the financial scholarshi NCQP for GC/MS and IR analyse BSTR for SEM images and SEM-EDS spectra; and FFCLRP for TG measurements.

References:
[1] Doumer, M. E. et al., JAAP 113, 434-443 (2015)
[2] Dorado, C., Mullen, C. A., Boateng, A. A. APCATB 162, 338-345 (2015) [3] Su, Y. et al. BIORTECH 295, 122243 (2020)

AgNPs/Zn(mim) supported nanocatalyst applied in the dehydrogenation of hydrogen-carrier molecules

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Hydrogen-carrier molecules are a form of chemical hydrogen storage and have been shown as a promising, safe, clean, and renewable alternative to meet the emerging need to replace fossil fuels emitting CO_2. Since such molecules are resistant to hydrolysis, an efficient catalyst is required. The main properties of Metal-Organic Frameworks (MOFs), such as high porosity, high surface area, as well as chemical and thermal stability, make them promising materials in terms of several relevant applications, including catalysis [1,2]. The biggest problem with metallic nanoparticles (MNPs) is their high surface energy, which causes agglomeration and catalytic deactivation [2]. To prevent the aggregation of MNPs, in this work, the ship-in-bottle method was used to synthe a heterogeneous catalyst composed of a MOF, Zn(Mim) with supported silver nanoparticles (AgNPs). This material was used in the conversion of small molecules, such as NH3BH3 (AB) and NaBH4 (B), into hydrogen. The catalytic activity was verified by comparing with control experiments with AgNPs and support [Zn(Mim)] separately. It was shown that the produced nanocomposite, AgNPs/Zn(Mim), presented catalytic improvement compared to the support and AgNPs . Despite the low silver content (0.18 wt%), interesting results of hydrogen generation rate and rotation frequency were obtained for AB (2286.4 mL.min-1.gcat-1 and 10 molH_2.molcat-1.min-1) and B (3075.41 mL.min-1.gcat-1 and 13.49 molH_2.molcat-1.min-1) hydrogen carriers. Furthermore, the materials were also characterized by SEM, ICP-OES, FTIR, XRD, TGA/DTA, and BET.

Acknowledgements:
The authors gratefully thank for scholarships granted: CNPq (141076/2019-4), CAPES (88887.643078/2021-00), and FACEPE (IBPG-0477-3.03/19)

References

[1] Gao, D. et al. Chemical Engineering Journal 361, 953-959 (2019).
[2] Alhumaimess, M. S. Journal of Saudi Chemical Society 24, 461-473 (2020).

Analyses of $BiVO_4/Fe_2O_3$ heterojunction based photoanode for photoelectrochemical water splitting

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Photochemical process can be used to solar energy storage in the form of hydrogen or low-carbon fuels and are promising systems for chemical energy conversion [1]. For this reason, researches have been carried out to find cheap and efficient materials for these systems. In this context, this work shows the synthesis and characterization of $BiVO_4$(BV), Fe_2O_3(F), and $BiVO_4$-Fe_2O_3(BVF) compounds and analyzes their performance for photoelectrochemical water splitting. The compounds were prepared by precipitation method [2] and subsequently, were deposited, as films, on a conductive FTO (fluorine-doped tin oxide) substrate and tested photoelectrochemically by doctor blade process. The materials were analyzed by X-ray diffraction and the results showed that BV/BVF and F have the crystalline phases of monoclinic and hematite, respectively. Agglomerates of different particles were observed by scanning electron microscopy. The photoelectrochemical tests were carried out between -0.55 and 0.85 V at a scan rate of 50 $mV.s^{-1}$. As results, the current densities obtained for BV and BVF were 6.61 and 14 $\mu A.cm^{-2}$, respectively, up to 1.23 V vs. RHE (reversible hydrogen electrode) under a light source, in contrast to F, without any photoelectrochemical response. For the photoelectrochemical tests, when BV and F were mixed, there was an improvement in the response for hydrogen gas production. This phenomenon occurs because the excited photoelectrons move from the conduction band of the BV to the F, and at the same time, they move from the valence band of the F to the surface of the BV to oxidize the water. Therefore, the results showed that is possible use BV and F for hydrogen production using a photochemical process.

Acknowledgments:

This work was supported by the CAPES, CNPq, Fundação Araucária, FINEP and PRPPG/UNILA.

References:

[1] L. Montañés et al. Catalysts, 11(10), 1244 (2021).

[2] Y.-l. Li et al. Materials and Design, 187, 108379 (2020).

Analysis of Magnetic Circuit Performance for Thermomagnetic Devices

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Devices based on the magnetocaloric effect and magnetic phase transition require a source of magnetic field to operate. Permanent magnets remain the primary source of magnetic field in magnetic refrigerators and thermomagnetic motors. However, the performance of these permanent magnets, is evaluated using the Λ_{cool} parameter, developed to evaluate permanent magnets used in refrigeration systems. Therefore, the objective of this work is to perform a parametric and comparative analysis of a C-type and double C-type permanent magnet based on a common parameter to any arrangement of magnets: the figure of merit M*. The analysis was conducted by computational modeling and simulation using a Multiphysics software. Different soft magnetic materials were used in the simulations and the best-performing was used in the magnetic circuit of the C-type permanent magnet to evaluate the action of the magnetic force on a set of gadolinium plates. As a result, it was observed that the soft magnetic material has no significant impact on M*. The same observation can be made regarding the number of gaps, as long as more magnetized blocks are employed in the arrangement. Furthermore, a higher magneto-static force density on a magnetocaloric material can be achieved with larger gaps.

Analysis of Mechanical Parameters Effects onNanofluids Preparation Using 2-step Method

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Nanofluids are composed of a base fluid and a fraction of dispersed nanoparticles to enhance the base fluid characteristics such as thermal conductivity, stability, and tribology aspects. The enhancement level of a nanofluid is related to nanoparticle stability, which can be made through chemical or mechanical processes. The chemical stability process is made up of interaction between the base fluid and nanoparticle materials using surfactants, while the mechanical stability process relates to the preparation methods and their characteristics. The 2-step method is the most common nanofluid preparation method, and it is greatly affected by mechanical parameters. The stirring speed, stirring time, and sonication features (exposure time, energy, and amplitude), represent some of the relevant mechanical parameters that may affect the nanofluid stability. In this work, an Al2O3-H2O nanofluid was investigated aiming to depict the mechanical parameters of the nanofluid's stability. For this purpose, zeta potential, dynamic light scattering (DLS), and ultra-violet visible spectroscopy (UV-vis) measurements were performed. The results showed that stirring parameters have a small to medium effect on nanofluid stability, while sonication parameters have a medium to high impact on stability. The stirring parameters tend to decrease the nanoparticle agglomeration level. On the other hand, the sonication parameters improve the level of nanoparticle dispersion. Thus, it was shown that interesting stability levels can be reached by combining both stirring and sonication processes properly.

Assessment of the blends of babassu residual cake and rice husk for briquette production

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The growing demand for energy and concern about the environmental impacts generated by the excessive use of fossil fuels has reinforced the search for renewable sources, such as biofuels. The present work aimed to evaluate the production of briquettes from the residual cake obtained from the extraction of babassu oil in a mixture with rice husk at proportions of 25%, 50%, and 75%. The authors assessed moisture content (M), volatile matter (VM), ash (A), fixed carbon (FC), and the Higher Heating Value (HHV) of briquette of the residual babassu cake (RBC) and the briquettes of the blends RBC/rice husk (RH). According to the results, the RBC briquette presented 3.60% of M, 85.28% of VM, 3.72% of A, 7.40% of FC, and an HHV of 22.25 MJ/kg. The briquettes of the blends presented the following results - Moisture: 9.99% for RBC(75%)/RH(25%), 9.54% for RBC(50%)/RH(50%), and 8.19% for RBC(25%)/RH(75%); Volatile Matter: 82.71% for RBC(75%)/RH(25%), 79.03% for RBC(50%)/RH(50%), and 75.31% for RBC(25%)/RH(75%); Ash: 6.75% for RBC(75%)/RH(25%), 10.15% for RBC(50%)/RH(50%), and 13.21% for RBC(25%)/RH(75%); Fixed Carbon: 5.50% for RBC(75%)/RH(25%), 1.28% for RBC(50%)/RH(50%), and 3.28% for RBC(25%)/RH(75%), and Higher Calorific Value: 19.22 MJ/kg for RBC(75%)/RH(25%), 17.94 MJ/kg for RBC(50%)/RH(50%), and 17.26 MJ/kg for RBC(25%)/RH(75%). Given the above, it concluded that the residual babassu cake mixed with rice husk has the potential for use as a direct source of energy generation and as a means of improving the performance of other biomass for energy purposes [1].

Acknowledgments: This work was supported by CNPq (308280/2017-2, 313647/2020-8); FUNCAP (PS1-00186-00255.01.00/21); FINE and CAPES (Finance Code 001).

References:[1] SILVA, C. B. S. ; SOUSA, B. A. ; NUNES, J. V. S. ; MALVEIRA, J. Q. ; MARQUES, R. B. ; DAMASCENO, L. B. ; BRAGA, E. F. ; NASCIMENTO, T. L. ; BERTINI, L. M. ; MACIEL, M. A. M. and RIOS, M. A. S., PROCESSES vol. 11, 585-600, (2023)

Barocaloric effect evaluation in thermoplasticelastomer

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Search for greater energy efficiency in cooling systems has driven the development of innovative technologies. In this context, solid-state refrigerator stands out as a promising alternative of new thermal exchange device, especially in systems based on i-caloric effects. Despite the great potential, few materials have already been tested for the barocaloric capacity, a specific type of i-caloric effect in response of applying pressure over a material. Therefore, this work aims to evaluate the barocaloric effect in Thermoplastic elastomer (TPE), a widely used material in different areas of the industry, which is capable of being easily processed with different types of additives. The TPE was processed by counter-rotating twin screw extrusion at 60 rpm in a single temperature of 190 °C and characterizedby FTIR, DSC, Young's modulus tests and Shore hardness. Samples were molded in a cylindrical geometry at temperature of 150 °C, and its barocaloric effect were evaluated bya calorimeter with direct measurement of temperature variation, obtaining the value of 11 Kat a pressure of 218 MPa. These values are comparable to the observed in polyurethane and others elastomeric materials [1], thus having a potential for application in refrigeration systems.

Acknowledgements:
The Author thank the graduated program in Mechanical Engineer/ State University of Maringa (PEM/UEM). This work was supported by CNPq.

References:

[1] J. R. Bocca and S. L. Favaro, Polym Test, vol. 100, p. 107251, 2021.

Carbon nanotubes/MOF nanocomposites prepared by Pickering emulsion method for electrodes of supercapacitors

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Energy storage devices are required for home and vehicle use. Lithium-ion batteries and supercapacitors are widely used, but they are not efficient enough to power large systems and still encounter certain limitations for use in automobiles and pulsed electronic devices. Of many materials, Metal-Organic Frameworks (MOFs) are promising because they form by self-assembly, have low density, high surface area and adjustable pore [1]. To improve electrochemical performance, carbon nanotubes (CNTs) were incorporated into MOFs, chosen for their large surface area and good electrical conductivity [2]. Thus, the composites were generated by the Pickering emulsion method, one from a Zinc Metal-Organic Framework coordinated with benzenecarboxylate, isophthalic acid and benzimidazole ligands, and another composite from a copper MOF coordinated with a benzenecarboxylate, isophthalic acid. After electrochemical analysis, the ZnMOF@CNT composite showed only capacitive behavior, resembling a supercapacitor with 4.94 F/g. The CuMOF@CNT composite showed capacitive and faradic behavior, resembling a pseudocapacitor.

Acknowledgements: The authors thank the FACEPE (IBPG-0609-3.03/20), and CAPES (88887.685516/2022-00) for the scholarships granted.

References: [1] S. Sundriyal, H. Kaur, S. K. Bhardwaj, S. Mishra, K. H. Kim, A. Deep, Coord. Chem. Rev. 2018, 369, 15-38.

[2] M. Wilamowska, M. Kujawa, M. Michalska, L. Lipińska, A. Lisowska-Oleksiak, Synth. Met. 2016, 220, 334-346.

Catalytic Reactions to a pre-treatment of biomass from urban waste

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The use of biomass as a precursor in synthesizing higher value-added products has grown in the research scenario focused on green chemistry. In this way, studies focus on treating raw biomass to obtain smaller molecules. When placed in an acid medium, the polymers are broken because the ester group that links the molecules to each other reacts with the medium. This work aims to use HBEA 25 zeolites and sulfonated biochar from coffee to catalyze this depolymerization process. [1][2]Zeolite HBEA 25 had its structure analyzed by SEM and FRX. Therefore, was possible to prove its crystalline structure, and by the BET method, it was possible to assess that it has a specific area of 488 m2/g. Total acidity obtained by TPD-NH3 showed a value of 0.575 mmol/g. Concerning, biochar acidity analysis was carried out by bench titration and obtained concentrations of H+ 0.004810. This sulfonated carbon from coffee grounds showed acidity averaging 0.9611 mmol/g. SEM images and IR analysis confirmed and polycyclic structure. As a future perspective of this project, there is the analysis by HPLC and Capillary Electrophoresis of the reaction made between these materials and the biomass to know the conversion of the response, in addition to looking for new catalytic materials and also to elaborate studies for reuse techniques of these materials. Acknowledgements: Faperj, UFF, Capes, Recat, Lamar Caipe References:1. FALEEVA, Y. M.; LAVRENOV, V. A.; ZAICHENKO, V. M. Investigation of plant biomass two-stage pyrolysis based on three major components: cellulose, hemicellulose, and lignin. Biomass Conversion and Biorefinery, p. 1-11, 2022. 2. T.M. Lima, C.G.S. Lima, A.K. Rathi, M.B. Gawande, J. Tucek, E.A. Urquieta-González, R. Zbořil, M.W. Paixão, R.S. Varma, Magnetic ZSM-5 zeolite: a selective catalyst for the valorization of furfuryl alcohol to γ-valerolactone, alkyl levulinates or levulinic acid, Green Che

Cerium, molybdenum and cerium-molybdenum oxides obtained by the Pechini method using glycerol as a polyol for application in fructose conversion

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The development of selective and efficient heterogeneous catalytic systems for converting biomass into products of industrial interest is the focus of several studies [1]. In this work, oxides of cerium (Ce100), molybdenum (Mo100) and a mixed oxide based on cerium and molybdenum (CeMo25: with 25% molybdenum) were synthed by the Pechini method, using glycerol as a polyol, and applied in the conversion of fructose. The textural and structural properties of the catalysts were elucidated by FTIR, XRD, TG/DTG, DRS, nitrogen physisorption analysis (BET and BJH), and FTIR using pyridine as a probe molecule. The X-ray diffractograms showed Ce100 had a cubic fluorite-type structure and Mo100 an orthorhombic phase. For CeMo25, the formation of cerium molybdate was identified [2]. CeMo25 showed higher thermal stability and an adequate modulation of acidic sites compared to Mo100. Furthermore, CeMo25 exhibited higher catalytic activity (91.8%) compared to Ce100 (36.8%) already within 3 reaction hours. The mixed oxide exhibited lower formation of insoluble materials and selectivity to products of the retro-aldol route and 5-HMF. The results obtained showed that the main factor influencing fructose conversion is the acid sites present in the catalysts [1]. CeMo25 was used in 4 cycles, with no leaching or significant decrease in the activity observed. All observed characteristics suggest that the synthed oxides are promising for converting biomass into valuable chemicals.

Acknowledgements:

CNPq, CAPES, FAPEAL, UFAL, PPGQB, IQB, GCAR.

References:

[1] T.V. Dos Santo; D.B.A. Pryston and G.C. Assis, Catalysis Today 379., 62-69 (2021).

[2] T.Ito; K. Sunada and T. Nagai, Materials Letters 290., 129510 (2021).

Chemical characterization and evaluation of the electrocatalytic water oxidation reaction promoted by Copper-Prussian blue after termal decomposition

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The following work aims to evaluate the efficiency of different types of Prussian blue catalysts, $[Cu[Fe(CN)_6]_{0.5}*nH_2O]$, in the water oxidation reaction. The evaluated catalysts were prepared by thermal decomposition at different temperatures (60, 140, 400, and 900°C), and XRD, Raman, and FTIR techniques were used for their characterization. To evaluate the performance of the catalysts, their electroactive Area, the Onset value and the Tafel slope were calculated. The results of the chemical characterization showed that the material heated at 60°C and 200°C maintained the crystalline structure and the characteristic bands of a Prussian blue analog, which is related to the cyanide (ν(-CN)) stretching centered at 2100-2200 cm^{-1} in both the Raman and infrared spectra.[1] When the temperature reached 400°C and 900°C, bands characteristic of copper oxide and mixed copper-iron oxide were observed.[2] The catalysts with the highest water content (60 and 200°C) showed lower performance in the water oxidation reaction than the catalysts prepared at 400 and 900°C, which showed the best performance and the highest number of active sites. From these results, we observed that thermal treatment can produce different catalysts using the same precursor. Moreover, the water content of the catalysts modified their performance with respect to the water oxidation reaction. Further studies on the effect of water and surface area on these catalysts under mild conditions are essential to better understand the water oxidation mechanism and to advance the development of new catalysts.

Acknowledgements:

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior- Brasil (CAPES) - Finance Code 001. The authors are grateful to the CAPESP, CNPq (308203/2021-6), FAPESP-2017/11986-5, 2021/05976-2

References:

[1] Priscilla J. Zambiazi et. al. Dalton Transactions 49,16488, 2020.

[2] L. S. Medletshe, et. al. Nanomaterials 9(8), 1140, 2019.

Computational Study of the Intermolecular Interactions of Ionic Liquids Derived from the Imidazolium

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Ionic liquids play a range of applications, with CO_2capture being one of them [1]. Such applications have motivated several studies with the goal of elucidating the behavior of its properties [2]. Ionic liquids based on imidazolium cation have characteristics that aid in the CO_2capture process, which in turn are related to their intermolecular interactions [3]. The objective of this work is to understand how the different substitutions in imidazolium cation affect its intermolecular forces and assist in the capture of CO_2. Molecular dynamics calculations were performed for 14 ionic liquids based on imidazolium cation. RDF, SDF, and MSD calculations were performed in the system structure containing 200 pairs of ionic liquid. After the analysis of the molecular trajectories of these structures, the representative structures of the system were taken and calculations of NCI and DFT were performed. The equilibrium distance between cation and anion remained the same in general. Van der Waals interactions proved to be important for the formation of the ionic pair cluster and interaction free energy proved to be an important driver in the formation of the ionic pair. The correlation between Henry's constant and the value of long-range interaction energies in bulk has shown that they can provide valuable information for the prospection of new IL with CO_2capture potential.

Acknowledgements

The authors gratefully acknowledge the financial support from INCT-INAMI, CNPq, CAPES, FAPESQ-PB, PRONEX-FACEPE, FADE-UFPE, FINEP, and the physical structure and computational support provided by UFPB, CENAPAD-SP, NPAD/UFRN, and Supercomputer Santos Dumont. This study was financed by UBEA/PUCRS-FADE/UFPE-UFPB through grant № 317/22 (UBEA/PUCRS) and grant № 01/2022 (FADE/PUCRS).

References:

[1] . Cheng, J. et al. Int. J. Mol. Sci. 23, (2022).
[2]. Aghaie, M. et al. Renew. Sustain. Energy Rev. 96 (2018).
[3]. Shariati, A. et al J. Supercrit. Fluids 29, 43-48 (2004).

Copper-based thin films deposition by magnetron sputtering for photoelectrochemical water splitting

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One of the main challenges in the development of photoelectrochemical (PEC) water splitting (WS) devices is the formation of active, stable and low-cost photocathodes. CuO is considered a promising material as a photocathode for PEC reactions, but generally suffers from low stability because the oxidation state of copper is easily changed. Based on our experimental findings utilizing various techniques such as XRD, Raman, and XPS, we demonstrated that CuO was formed on FTO substrate (Fluorine-doped Tin Oxide). Our results by profilometry indicates that the film has a thickness of 200 nm. The UV/Vis spectra combined with Tauc Plot reveal a bandgap value of 2.2 eV. As expected, preliminary tests of PEC measurements demonstrate a photocurrent density of 0.5 mA/cm2 for the pristine CuO film. However, due to its low stability, we employed a strategy involving the deposition of an ultra-thin layer of Si and SiO2 on the surface of film. This was done to minimize the CuO reduction sites to Cu metal and enhance the stability of the film. Additionally, we employed very thin layers of BiVO4 and TiO2 to increase the chemical stability of the films.

Acknowledgements:The authors are grateful to CAPES (88887.714087/2022-00) and FAPESP (Grant 2017/18716-3, schoolarship 2019/18724-1) for funding this research project. We also thank the National Center for Energy and Materials Research (CNPEM) for the experimental facilities (INTERCOVAMEX H2 sputtering system).

References:

[1] R. V. Gonçalves, et al., in Nanoenergy, Springer International Publishing, pp. 107-140 (2018).

[2] W. S. Rosa et al., in ACS Aplied Materials and Interfaces, pp. 22858-22869 (2022).

[3] A. dos Santos Corrêa, et al., in Energy Advances, pp. 123-136 (2023).

DEVELOPMENT OF BIVO4 PARTICLES WITH EXPOSED FACETS TO BOOST LIGHT-DRIVEN CO2 REDUCTION INTO SOLAR FUELS

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The dependence on the production of energy from fossil fuels such as oil, natural gas and coal generates the emission of large amounts of greenhouse gases, which are spontaneously emitted into the environment, causing dangerous environmental effects such as global warming, which affect the lives of millions of people. One strategy to minimize the environmental crisis is artificial photosynthesis which, through the use of semiconductor materials and sunlight, can reduce CO2 and convert it into products of high chemical value. One of the widely studied semiconductors is Bismuth Vanadate (BiVO4), a material with unique optical, electronic, and structural properties that make it an interesting candidate for the production of solar fuel. In the present work, BiVO4 particles were synthed by the hydrothermal method controlling the pH of the aqueous solution. The XRD and Raman results indicated the formation of a monoclinic phase. The MEV showed that the particle increased with increasing pH, reaching a well-shaped and exposed facete with pH equal to 1. The UV-Vis spectra showed that the pristine BiVO4 have a band gap of approximately 2.3 eV. The photocatalytic CO2 reduction using BiVO4 with well-exposed facet presented unique activity to convert to CO and CH4 products. After 5h of reaction in an aqueous medium, BiVO4 /pH 1 produced 0.213 µmol and 0.022 µmol of CO and CH4, respectively. In order to improve the photocatalytic activity, Ruthenium nanoparticles were deposited on the surface by the chemical impregnation method in different concentrations, as a result it was obtained that for BiVO 4 /0.05%wt Ru/pH 1, the amounts of CO and CH4 were 0.462 µmol and 0.132 µmol, respectively, showing a significant increase at 117% CO and at 500% CH4, effectively increasing their photocatalytic activity.(1,2)

Acknowledgements

FAPESP and Shell/RCGI

References

1. Zhao, y. et al. Cryst. Growth Des. 2017, 17, 6, 2923-2928
2. Han, q. et al. ACS Appl. Mater. Interfaces

Development of SiO2-BaF2 anti-reflective coatingapplied as solar glass

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In this study, we present a methodology for developing a broadband antireflective coating deposited on a glass substrate, which incorporates porosity in the films. We evaluate the film's application as solar glass in covering a monocrystalline silicon solar cell [1], and we examine the increase in transmittance of the film compared to the glass substrate. Additionally, we investigate the influence of the films on the photovoltaic parameters, such as the fill factor (FF) and power conversion efficiency (PCE). We synthed SiO2-BaF2 by sol-gel and dip-coating deposition, where we varied withdrawal from 0.5 mm/s to 4 mm/s, followed by thermal treatment at 100°C for 1 h. We characterized the films by analyzing their transmittance and reflectance spectroscopy, and scanning electron microscopy. We also performed electrical characterization of the solar cell covered with the different films and the glass substrate to obtain the parameters of short circuit current density (Jsc) andopen circuit potential (Voc), which allowed us to determine the FF and PCE. The results of transmittance in the visible region (400-800 nm) show that the films with different withdrawal had the following transmittance: 0.5 mm/s with 92.9%, 1 mm/s with 93.9%, 2 mm/s with 95.9%, 3 mm/s with 95.7%, 4 mm/s with 96.0%, and the glass substrate with 91.8%. The film shows an approximate 4.5% improvement in transmittance in the visible region compared to the glass substrate. The increase occurred due to the insertion of porosity, which is related to the in-situ formation of hydrofluoric acid (HF). Porosity contributed to the graded index of refraction in the SiO2-BaF2 film, and these conditions influenced reflectance suppression. And we evaluated the PCE and FF of the solar cell with the device covered with the glass substrate and with the best transmittance film.

Acknowledgement:
CAPE CNPq; CCAF; CDMF; PPGFí UFPEL.

References:

[1] K. C. S. Reddy, et al., Solar Energy, 190 (2019), 119-125.

Development of sputtered BiVO4-CuO Tandem cells for bias-free solar water splitting.

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Photoelectrochemical (PEC) water splitting is a promising technique for converting solar energy into green hydrogen, addressing global energy and environmental issues[1].Developing Tandem PEC deviceswith stable metal oxides has recently attracted attention for cleaving H_2O moleculesinto H_2 using only solar energy[2]. Herein, $BiVO_4/FeNiO_x$ and CuO photoelectrodes were synthesized via magnetron sputtering to perform oxygen and hydrogen evolution reactions (OER and HER, respectively) in a simple Tandem PEC cell. Our $BiVO_4/FeNiO_x$ photoanode showed excellent PEC performance and chemical stability for OER, achieving a photocurrent density of +1.22mAcm-2 and a charge transfer efficiency of 96% at the water oxidation potential. In contrast, the all-sputtered CuO photocathode exhibited a modest PEC performance for HER, with an onset potential at 1.03V vs. RHE (reversible hydrogen electrode) and photocurrent density of -0.35mA.cm-2 at +0.40V vs. RHE. In this work, we also investigated the energy diagrams of the photoelectrodes under working conditions for PEC water splitting. Our results demonstrated that the photogenerated holes (electrons) at the $BiVO_4/FeNiO_x$ (CuO) film have enough energy to perform the OER (HER) without external bias. Conclusively, our $BiVO_4/FeNiO_x$-CuO Tandem PEC cell produced a stable operating photocurrent density of ~50μA.cm-2 under zero-bias and AM 1.5G illumination for at least 1000 seconds, evidencing the occurrence of bias-free solar water splitting reactions.

Acknowledgements:
The authors are grateful to CAPES and FAPESP (Grant 2017/18716-3, 2019/18724-1), RCGI - Research Centre for Greenhouse Gas Innovation, hosted by USP and Shell Brazil, for funding this research project.

References:

[1] Li, Z. et al. Energy Environmental Science, v. 6, n. 2, p. 347-370, 2013.
[2] Chen, Y. et al. ACS Energy Letters, v. 5, n. 3, p. 844-866, 2020.

Development of Tandem BiVO4-LaFeO3 cells for solar hydrogen production through artificial photosynthesis without external field application

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The development of solar energy technologies is crucial to achieving the sustainable development of human society. To solve the problems of environmental pollution and fossil fuel shortage, recent technologies have incorporated innovative solutions [1, 2]. This study aims to investigate a Tandem cell composed of thin films of semiconductor materials, BiVO4 and LaFeO3, to produce solar H2 from water and sunlight without external polarization. The films were deposited on fluorine-doped tin oxide (FTO) substrates using RF-Sputtering techniques and ceramic targets processed by the research group [2,3]. XRD and Raman spectroscopy confirmed the monoclinic and pure phases of the LaFeO3 and BiVO4 films, respectively. The thickness of the films was determined by profilometry, indicating 80 nm for BiVO4 and 100 nm for LaFeO3 films. UV/Vis spectra demonstrated a bandgap value of 2.5 eV for BiVO4 and 2.1eV for LaFeO3. PEC measurements showed a photocurrent density of 1.1 mA/cm2 for BiVO4 and 100 □A/cm2 for LaFeO3 films. Therefore, the low photocurrent density of LaFeO3 led to the deposition of a layer of BiVO4 over the LaFeO3 film to increase the photocurrent density. Furthermore, a layer of NiFe was deposited over BiVO4 to increase its stability. These results provide crucial information about the properties and performance of BiVO4 and LaFeO3 films and suggest promising applications in solar energy conversion such as tandem cell systems.

Acknowledgements:
The authors most gratefully acknowledge the financial support FAPESP, RCGI-USP and Shell Brazil, CNPq (Grant 152738/2022-3).

References:
[1] O. Coddington, et al. Bull. Amer. Meteor. Soc., 163 (2016), pp. 1265-1282.
[2] W. S. Rosa, et al., ACS Appl. Mater. Interfaces, 2022.
[3] CORREA, ANDRESSA S., et al. Energy Advances, v. 1, p. 1-10, 2023.

Diamond-like silicene nanosheets surface-doped:physical properties and potential applications

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Two-dimensional (2D) materials have been widely investigated since graphene was synthed in 2004. One of the most fascinating characteristics of these materials is the possibility of doping and/or functionalizing them with several and different kinds of atoms, making their properties vary considerably [1]. Consequently, it is of great interest to investigate and understand the physical properties of doped 2D structures since they are potential candidates for applications in several and interesting areas. Whithin this panorama, we explored the properties of diamond-like silicene nanosheets, namely bilayers and trilayers, functionalized with boron, nitrogen, aluminum and phosphorus atoms [2]. In particular, the three-layer doped silicon systems present scarce literature and, in this work, we found four novel nanosheet systems, named ABC-Si_4B_2, ABC-Si_4Al_2, AA'A''-Si_4P_2, and ABC-Si_4P_2, with interesting physical properties and promising applications. Our investigation provides these systems' structural, thermodynamic, dynamic, elastic and electronic properties. Analyzing the electronic structure of these materials, we found two distinct groups of dynamically stable nanosheets: metallic and semiconductors systems. Furthermore, we discuss the possibility of using these nanosheets in alkali metal ion batteries (AMIBs), van der Waals heterostructures, UV light devices and thermoelectric materials. Finally, we already demonstrate that the ABC-Si_4Al_2system is a promising candidate for AMIBs anodes, mainly for sodium and potassium ion batteries [3].

Acknowledgements: This investigation was partially supported by Brazilian agencies CNPq, CAPES and FAPESP (22/10095-8).

References:

[1] B. Ipaves, J.F. Justo, and L.V.C. Assali, J. Phys. Chem. C 123, 23195 (2019).

[2] B. Ipaves, J.F. Justo, and L.V.C. Assali, Phys. Chem. Chem. Phys. 24, 8705 (2022).

[3] B. Ipaves, J.F. Justo, and L.V.C. Assali, Mol. Syst. Des. Eng. 8, 379 (2023).

Effect of porosity parameters and physicochemical properties of different microporous and hierarchical zeolites on the CO_2 adsorption process

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In this study, the effect of the application of microporous zeolites with different pore systems (one-dimensional ZSM-23, two-dimensional ferrierite and three-dimensional LTA) and physical-chemical properties in relation to their hierarchical zeolites (micro-mesoporous) derived from the adsorption process of CO_2at 30 °C under atmospheric pressure. The zeolites were characterized by X-ray diffraction, energy dispersive X-ray, nitrogen adsorption-desorption and temperature-programmed desorption of carbon dioxide. The synthesized microporous zeolites (ZSM-23, FER and LTA) presented physicochemical characteristics similar to those described for their structures in the literature. The post-synthesis treatment via desilication (in the case of zeolites ZSM-23 and FER) and the use of sodium glutamate (in the synthesis of zeolite LTA-Glut), allowed obtaining hierarchical zeolites (micro-mesoporous). The hierarchical zeolites showed a higher CO_2adsorption capacity (between 0.3 $mg_{CO2}/g_{Adsorbent}$to 13.7 $mg_{CO2}/g_{Adsorbent}$) than the microporous zeolites (between 0.6 $mg_{CO2}/g_{Adsorbent}$and 10.4 $mg_{CO2}/g_{Adsorbent}$) due to the simultaneous action of their higher area and volume, concentration and strength of the basic sites and the presence of mesoporosity, with a strong dependence on the increase in the adsorption capacity for zeolites with more open pore systems (LTA three-dimensional pore system >FER two-dimensional pore system >ZSM-23 one-dimensional pore system).

Acknowledgments:
This work was supported by CNPq, UFAL, UFBA and UFU

Effects of La Content in Ceria-Lanthana Thin Films prepared by Pulsed Laser Deposition

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Solid oxide electrochemical reactors are a promising alternative for challenging chemical reactions, such as oxidative coupling of methane (OCM)[1], which convert methane to C^{2+}products. Cerium oxide is a commonly studied oxide membrane material for its high ionic conductivity in the temperature range ideal for conversion reactions (600-900°C), particularly when doped with rare earth cations like Gd, Sm, or La. Recent studies have shown $La_2Ce_2O_7$as a promising OCM catalyst[2]. This study investigates the structural properties of $La_xCe_{(1-x)}O_{2(1-x/2)}$thin films, prepared using pulsed laser deposition, with varying stoichiometry. XRD results indicate textured film growth with preferential [100] orientation of cubic crystalline fluorite for low La content (x≤0.5), while x>0.5 had a random orientation. La doping expanded the fluorite structure, increasing lattice parameters from 5.42 Å for x=0 to 5.69 Å for x=0.7. UV-Raman spectroscopy showed that La content x=10 caused a more pronounced balance between $Ce^{4+}$and Ce^{3+}than pure ceria samples. Impedance revealed a direct relationship between La content and resistance, indicating lower resistance with lower La content. These findings could lead to using Ce-La oxides thinfilms as catalysts for OCM in electrochemical reactors based on ceria.

Acknowledgments:
The authors acknowledge to CNPq (142538/2020-5) and Center for Innovation on New EnergiesShell (ANP)/FAPESP (2017/11937-4). F.C.F., D.Z.F., and A.S.F. are CNPq fellows.

References:

[1] I. Garagounis, V. Kyriakou, C. Anagnostou, V. Bourganis, I. Papachristou, and M. Stoukides, "Solid electrolytes: Applications in heterogeneous catalysis and chemical cogeneration," Ind. Eng. Chem. Res., vol. 50, no. 2, pp. 431-472, 2011.

[2] M. Coduri, S. Checchia, M. Longhi, D. Ceresoli, and M. Scavini, "Rare earth doped ceria: The complex connection between structure and properties," Front. Chem., vol. 6, no. OCT, pp. 1-23, 2018.

Electrocatalytic activity of CoFe Prussian blue analogues and their calcination product towards the oxygen evolution reaction in neutral and alkaline media

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Water splitting is one of the most promising methods to produce renewable energy, nevertheless the oxygen evolution reaction (OER) involves a complex multistep proton-coupled electron transfer process, meaning this half reaction is kinetically sluggish and requires a large overpotential. Therefore, an electrocatalyst must be used to overcome the thermodynamics and kinetics requirements [1,2]. Prussian blue analogues (PBA) are considered remarkable catalysts for water oxidation in neutral medium and they have attracted increasing attention. In addition, PBA can also be used as a template to generate oxides by calcination, like the bimetallic spinel oxides which have emerged as outstanding candidates for oxygen evolution reaction catalysts [1,3]. Thus, this work aimed to investigate the electrocatalytic activity of $Co_3[Fe(CN)_6]_2$ and $Co_4[Fe(CN)_6]_2$ films and the oxides derived from their calcination at 500°C towards OER in neutral and alkaline media.To carry out this study, CoFe Prussian blue analogues films were deposited on fluorine doped tin oxide (FTO) coated glass by using different methods (drop-casting, electrochemical and spray coating). These films were afterward calcined at 500°C and electrochemical measurements were made with all of them in 0.5 M KNO3, 0.1 M KOH and 1 M KOH solutions. The results demonstrated that the calcined films are better catalysts for oxygen evolution reaction than CoFe PBA. Besides, the oxides showed to be significantly more efficient in alkaline medium and their electrocatalytic activity is affected by the deposition method employed to prepare them.

Acknowledgements:

The authors are grateful to the CAPESP, CNPq (308203/2021-6), FAPESP-2017/11986-5, 2021/05976-2.

References:

[1] Lei, S.; et al, Applied Catalysis B: Environmental, 245, pages 1-9 (2019)

[2] Sagu, J.S.; et al, Electrochemistry Communications, 87, pages 1-4 (2018)

[3] Zambiazi, P.J.; et al, Dalton Transactions, 49, pages 16488-16497 (2020)

Electrochemical characterization and structuralanalysis of $CaSr_xCu_{3-x}Ti_4O_{12}$ ceramic oxide for supercapacitor applications

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In this work, we present a comprehensive analysis of the electrochemical and structural properties of $CaSr_xCu_{3-x}Ti_4O_{12}$(x = 0.00, 0.15, 0.30, 3.00) (CSCTO) ceramic oxide for its potential use as a supercapacitor material [1]. The CSCTO samples were synthesized using a solid-state reaction method and characterized using various techniques such as X-ray diffraction (XRD), scanning electron microscopy, and electrochemical measurements. The CSCTO samples were deposited as films on an FTO (fluorine-doped tin oxide) substrate, and the electrochemical performances were investigated using cyclic voltammetry. The crystal structure and phase composition of the ceramic oxide samples were analyzed using XRD. All samples had a perovskite-like crystal structure. The CCTO sample (x = 0.00) exhibited only the $CaCu_3Ti_4O_{12}$with a cubic phase, while the SCTO sample (x = 3.00) showed the $Sr_{0.75}Ca_{0.25}TiO_3$with a tetragonal phase, which is in agreement with previous studies [2]. The CCTO 0.15Sr and CCTO 0.30Sr samples presented a mixture of CCTO and SCTO phases with slight stoichiometric variations. The specific capacitance values were determined using cyclic voltammetry, and the results showed that the CCTO 0.15Sr sample exhibited the highest specific capacitance of 22.11 mF g^{-1}at a scan rate of 100 mV s^{-1}, followed by CCTO with 5.77 mF g^{-1}, CCTO 0.30Sr with 7.4 mF g^{-1}, and SCTO with 0.8 mF g^{-1}.

Acknowledgments:
This work was supported by the CAPES, CNPq, Fundação Araucária, FINEP and PRPPG/UNILA.

References:

[1] S. Maity et al. Journal of Solid State Chemistry, 269, 600 (2019).
[2] R. Ranjan and D. Pandey, Journal of Physics C: Solid State Physics, 13, 4239 (2001).

Electrochemical characterization of $BaZr_{0,4}Ce_{0,4}Y_{0,1}Gd_{0,1}O_{3-\delta}$ protonic perovskite thin film

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Rapid population growth and the effects of urbanization urgently demand new technologies for efficient and sustainable energy storage and conversion in order to replace energy sources from fossil fuels. Thinking about this scenario, fuel cells emerge as a promising alternative due to their high efficiency and flexibility regarding the use of fuels, in addition to low or no CO_2emissions. Solid oxide cells have greater chemical stability and greater durability when compared to other types of cells, but one of their limitations is their operating temperature (800 - 1000 °C), to solve this limitation the next generation of SOFC passed to study proton cells that have a high conversion rate at intermediate temperatures (500 - 700 ºC). Among the most studied solid oxide electrolytes are barium zirconate cerate perovskites, which is an ABO_3type perovskite[1]. In addition to the study of new compositions, there are new designs of materials and structures that allow optimizing the operating temperature. As the thickness of the electrolyte is a determining factor in the performance of the cell, since its ohmic resistance is directly related to electrolyte thickness, the advancement in cell technology has progressed towards smaller and smaller thicknesses using more advanced deposition techniques such as PLD (Pulsed Laser Deposition) [2]. In this work the aim is to compare and characterize by X-ray diffraction (XRD), scanning electron microscopy (SEM) and impedance spectroscopy (EIS), sintered pellets and thin film deposited by PLD.

Acknowledgements:
The authors acknowledge to CAPES (88887.720450/2022-00).

References:

[1] Medvedev D, Murashkina A, Pikalova E, Demin A, Podias A, Tsiakaras P. BaCeO3: Materials development, properties and application. Prog Mater Sci. 2014;60(1):72-129.
[2]Zhou Z, Nadimpalli VK, Pedersen DB, Esposito V. Degradation mechanisms of metalsupported solid oxide cells and countermeasures: A review. Materials (Basel). 2021;14(11). doi:10.3390/ma14113139.

Electrochemical properties of carbon-supported palladium nanostructures with different metallicloadings

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High-surface area carbon-supported palladium (Pd/C) nanostructures are of great importance for electrochemical energy conversion and storage devices, such as fuel cells, water electrolyzers, and electrochemical sensors. However, the influence of the amount of metal anchored to the support on the electrochemical properties is not yet understood [1]. In this work, the electrochemical response in 0.1 M H2SO4 of Pd/C electrocatalysts (prepared previously by a colloidal synthesis method) containing different mass fractions of metal over carbon was investigated using the cyclic voltammetry technique. The results showed that the amount of palladium oxide (PdO) formed on the Pd/C electrodes clearly depends on the metal-to-carbon ratio, where an increase in the metal mass fraction leads toa decrease in the coverage of PdO. In contrast, the ratio between the amount of hydrogen desorbed and adsorbed (Hdes/Had) from the electrode was found to increase with the metal loading. A ratio (Hdes/Had) < 1 can be attributed to a spillover o Had towards the carbon phase [2]. Therefore, our results suggest that such a spillover effect decreases with the increase in the metal loading likely due to a reduced metal-metal interparticle distance expected to occur as the metal loading is increased.

Acknowledgements:
Univesp (scholarship for EP); Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq, grant 476690/2013-7).

References:
[1] CIAPINA, E. G. et al. International Journal of Hydrogen Energy, 43 (37), 17748 (2018).
[2] KONDA, S.; CHEN, A. Materials Today, 19(2), 100 (2016).

Electronic structure characterization of supported-metal catalysts by XPS technique

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Supported-metal catalysts are used in the chemical industry from fine chemical production to petrochemicals and consist of metallic particles dispersed on the surface of a metallic oxide support with different geometrical formats [1-3]. Catalysts with metallic active phases are used for reforming reactions since they have a high capacity to cleave C-C bonds, an essential step for high conversion of hydrocarbons into hydrogen rich syngas. Techniques for catalysts processing from a predefined support usually employ three fundamental steps: increase of the surface area by chemical route; impregnation of the active phase; and heat treatment. Each step directly influences either chemical composition or electronic configuration of the active phases, where the latter property plays a key role on the catalyst activity. In this work, X-ray photoelectron spectroscopy was used to investigate the chemical properties of alumina supported metal catalysts, e.g., Ni, Pt, Cu and Y, obtained by chemical and physical methods. The chemical composition and electronic states were evaluated from measurements of high-resolution spectra for the following chemical states: Al 2p, Ni 2p, Pt 4f, Pt 4d, Cu 2p, Y 3d, O 1s and C 1s. The valence band was also analyzed from the density of states measured between -10 and 30 eV. Results show that the preparation methods as well as the thermodynamic and chemical conditions are fundamental steps to the growing of catalytic surfaces with a strong signal of electronic states at the zero of the energy scale, which is assigned to the valence of the non-oxidized metallic particles.

Acknowledgments:

Authors thank BMW Brazil Group (grant nº UFSC/2016/0110), Fundep Rota 2030 / Linha V (grant nº 27192.01.01/2020.01-00) and CNPq (grant nº 307408/2021-3).

References:

[1] M. E. Silva Junior et al., ACS Omega, 8, 6507 (2023).

[2] A. M. Santos et al., Vacuum, 200, 111042 (2022).

[3] S. Anil et al., International Journal of Hydrogen Energy, 47, 8177-8213 (2022).

Enhanced Electrochemical Properties of Screen-Printed Electrodes with Polyaniline-based Hybrid Materials with Reduced Graphene Oxide for applications in Biofuel Cells

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Hybrid materials of polyaniline (PAni) with 2% graphite-based materials, such as graphene oxide reduced with ascorbic acid (rGO AA) and graphene oxide reduced with hydrazine sulfate (rGO SH), were fabricated and applied to hand-made screen-printed electrodes (SPE)[1] as modified anodes for use in biofuel cells with ***Saccharomyces cerevisiae***[2]. The materials were characterized using scanning electron microscopy (SEM), Raman spectroscopy, and attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), followed by evaluation through cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The bioelectrochemical system was characterized and studied using CV, EIS, and polarization curve. Results showed that PAni-rGO AA and PAni-rGO SH increased the current intensity in CV results, maintained the PAni voltammogramprofile, and reduced charge transfer resistance (EIE). However, the current intensity obtained was higher for the electrode modified with PAni-rGO SH. The SPE/PAni-rGO SHwas successfully applied to bicompartimental biofuel cells, obtaining electrical current from the ***S. cerevisiae***.

Acknowledgment:
FAPESP 2021/08041-4, 2017/24742-7, CNPq, Capes.

References:
[1] G.M. de Araújo, M.A. Cardoso, L. Codognoto, C.M.A. Brett, F.R. Simões, ECS Adv. 2 (2023) 016504.
[2] G. Silveira, M. Ikegaki, J.M. Schneedorf, Green Chemistry Letters and Reviews 10 (2017) 32-41.

Enhancing hydrogen storage capabilities of graphene oxide via Mg based nanostructures

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The global search for sustainable and renewable energy sources has gained significant momentum in recent years due to increasing concerns about climate change and the need to transition towards a low-carbon economy. Hydrogen has shown up as a great alternative, and one example is its use in fuel cells, which has water as a chemical green product. Many industries, like aerospace and automotive, are heading in the direction of the Hydrogen Economy and the electrification of these sectors is becoming increasingly inevitable. One of the challenges is, however, how to store H_2 molecules for later usage. Therefore, designing materials to capture those molecules properly is a great area of interest. In this work, we study Hydrogen adsorption on graphene oxide (GO) decorated with Mg (GO-Mg) from first principles calculations via Density Functional Theory (DFT). Functionals with Van der Waals (VdW) interaction corrections were used to take them into account. To model GO we used Hydroxyl and Epoxy functional groups which, when in contact with the Mg atom, originate different nanostructures with interesting structural and electronic properties. We used an energy based approach to calculate the stability of each GO-Mg nanostructures and analyzed the charge distributions on the systems. We found that Mg modifies the GO structure and is energetically favorable to form. Furthermore, the ability to capture H_2 molecules is enhanced in GO-Mg compared to pure GO. Adsorption energies were enhanced from 0.08 eV to 0.26 eV, now being in the desired range of 0.15 - 0.3 eV, in order to release the molecules from storage in ambient temperature without needing too much energy. The results indicate that GO-Mg has potential applications in hydrogen storage.

Environmentally friendlier perovskite nanocrystals for photovoltaic applications

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The class of materials with perovskite crystalline structure has excelled in the fields of energy conversion since its unique properties have enabled the development as optoelectronic materials with unprecedented speed, however, the research on more environmentally friendly methods for their synthesis, and on less toxic materials of enhanced stability are still in the early stages. [1] Among the improvement possibilities, potential "greener" alternative solvents are an appealing direction for enabling industrial processes.[2] We proposed the synthesis of perovskite NCs investigating the compatibility of "greener" solvents in established CsPbBr3 perovskite NCs synthesis. Water and limonene were used as solvents during the procedure, and LARP (Ligand-assisted reprecipitation) wasused as methodology. Influences of changes in procedure and components, such as surfaceligands, precursors molar ratios and temperature use, in obtained materials were evaluated,as well as ways for deposition in TiO2 substrates, focusing in application in energy conversion.

Acknowledgements:

This work was supported financially by CAPES, CNPq, FACEPE. Also, the authors gratefully acknowledge to the Centro de Tecnologias Estratégicas do Nordeste (CETENE).

References:

[1] Hoang, MT., et al. Green Chem. 2020, 22, 3433.

[2] Cheng, J., et al. Nanomaterials, v. 13, n. 6, p. 991, 2023.

Enzymatic catalyst for valorization of domestic food waste as a renewable feedstock

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Sustainable feedstocks are an alternative to supply society's energy demands without causing any environmental harms [1]. Approximately 1.6 billion tons of food are discarded worldwide every year which is often dumped in landfills causing environmental problems [2]. Thus, the aim of this work is to develop an efficient enzymatic catalyst for the degradation of domestic food waste with energy purposes. Hydrolytic enzymes were selected through tests varying the concentration of their solutions between 8 and 30 U/mL to compose a pool for immobilization via adsorption on magnetic biochar. The reactions were carried out at 40ºC under agitation at 120 rpm and tap water. The samples consistedof food waste in the proportion of 65% carbohydrates, 17% lipids and 18% proteins. The determination of lipase (Rhizopus niveus) activity was performed by acidity determination method by titration with NaOH. To evaluate the activity of cellulase (Aspergillus aculeatus), pectin lyase (mix of Aspergillus and Trichoderma reesei) and glucoamylase (Aspergillus niger), the 3,5-dinitrosalicylic acid method was applied. Celullase showed the highest activity (97 g/L of reducing sugars) in comparison with blank (20 g/L). Pectin lyase and glucoamylase also presented higher concentrations of reducing sugars (41 g/L and 47 g/L, respectively). Lipase activity achieved an acidity value of 78% in the oily phase against the blank (20%). The same enzyme in the aqueous phase generated an increase of 196% in the concentration of fatty acids. In addition, no pH variation was observed during the reaction. The free enzymes demonstrated excellent activity in the degradation of kitchen waste even though they were in a medium with tap water and no pH control. The next step is to immobilize the enzymatic pool on magnetic biochar and evaluate its activity.

Acknowledgements:CAPES,PPGQ-UFRGS

References:[1]Mumtaz, M. et al. Fuel.315,122840(2022) [2]Ajay, C.M.; Mohan, S.; Dinesha, P. Waste Management.125,10-26(2021)

Evaluation of nanostructured Co-Prussian Blue as a potential catalyst for Water Oxidation Reaction

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It is widely known that humankind' s current energy matrix is sustained by fossil fuels due to their relatively lower prices in relation to cleaner energy sources, but the ever-growing effects of the carbon emissions in global warming are the major incentive to maneuver towards more sustainable and efficient alternatives. A potential solution for this predicament is hydrogen gas (H_2). Still, since only a very small portion comes from renewable sources, many researchers are trying to concoct cheaper catalysts for Water Splitting. Therefore, this work aims to develop an optimally nanostructured Cobalt Prussian Blue Analogue (CoPBA) catalyst for Water Oxidation Reaction. Carrying out a coprecipitation method using potassium hexacyanocobaltate(III) (0.6 mol) and cobalt(II) acetate (0.4 mol), we have been able to consistently prepare multiple different morphologies modifying specific parameters (Fig. 1) to obtain even smaller structures, such as i) Addition of sodium citrate (0.9 mmol) as coordination agent ii) Usage of CoPBA seed (10 mg) iii) Increasing aging time, ranging from 3 hours to 7 days iv) Reacting it with 5 mL of ammonium hydroxide (NH_4OH) to prepare hollow nanocubes. The characterization of the CoPBAs was done via spectroscopic (FTIR, Raman, and UV-Vis) and diffractometric (XRD) techniques and we are currently evaluating its catalytic behavior using various electrochemical methods (CV, LSV, SECV, and others). Initial overpotential (η), Onset potential, and Tafel slope (A) comparations have shown noticeable differences between amorphous CoPBA (η = 1456 mV, Onset = 1834 mV and A = 228 mV.dec) and its nanostructured counterparts: CoPBA-citrate (η = 1409 mV, Onset = 1722 mV and A = 155 mV.dec^{-1}), CoPBA-Seed (η = 1511 mV, Onset = 1781 mV and A = 153 mV.dec^{-1}) and Hollow-CoPBA (η = 1409 mV, Onset = 1722 mV and A = 155 mV.dec^{-1}).

EVALUATION OF THE BAROCALORIC EFFECT INMETAL-ORGANIC FRAMEWORK (MOF) OF THE PEROVSKITE TYPE - NICKEL

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Current refrigeration systems use refrigerant gases as their working principle, which are responsible for a large part of the greenhouse effect. The development of technologies with greater energy efficiency and aimed at reducing environmental impacts has led to the search for new materials to replace these harmful gases. The use of solid refrigerants that have i-caloric effects is an alternative. The effect is the change in temperature and entropy of the material when exposed to external some force field. The barocaloric effect (σb-CE) is characterized by the heating or cooling of materials when subjected to isostatic tension (pressure) variation, in addition, the σb-CE effect is highlighted because it has been little explored, but simple to perform, due to its practicality. of the application of mechanical stress compared to a magnetic or electric field [1]. Metal organic frameworks (MOFs) are an emerging class of porous materials, prepared through several synthetic routes, through coordination reactions between metal ions and/or clusters with organic ligands. Perovskite-type MOFs have aroused great interest due to their ferroelectricity and ferromagnetism properties. Furthermore, some organic-inorganic hybrid perovskites showed a high barocaloric effect that aroused interest in the study of these materials [1]. Thus, the study aimed to synthesize, characterize physico-chemically and investigate the σb-CE of nickel perovskite MOFs. After X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analysis, the sample synthesis proved to be efficient, producing well-structured MOFs. Furthermore, with the direct measurements of adiabatic temperature difference, the sample showed a high value of ΔTs = 3.3 K, showing its potential for application in solid refrigeration.

References:

[1] IMAMURA, William et al. i-Caloric Effects: a proposal for normalization. arXiv preprint arXiv:1806.07959, 2018.

Giant barocaloric effect in vegetable oils, submitted to moderate pressures

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Search for greater energy efficiency in cooling systems has driven the development of innovative technologies. In this context, there has been a significant increase in the search for new materials that exhibit the barocaloric (BC) effect. We can highlight for example the colossal barocaloric effect discovered in the higher n-alkanes (adiabatic temperature changes for eicosane (C20H42) as 38 K up to 218 MPa and predicted colossal isothermal entropy changes higher than 900 J kg-1K-1). Another important discovery was the mechanocaloric compressive effect on cork (tensions up to 218 MPa with giant values of temperature change up to 13.6 K and entropy change up to 101.3 J kg-1 K-1) because it is a natural, renewable and sustainable material. Driven by these discoveries, mainly the manifestation of the mechanocaloric compressive effect (which is the general case of the barocaloric effect, characterized with the application of a hydrostatic pressure) in a natural materials, this work aimed to investigate the BC in corn oil and coconut oil, a commercially available, highly processed, low cost material in addition to being a natural, renewable and sustainable material. To measure the BC effect, 2.5ml of the oil was used, placed in a cavity with a diameter of 12 mm. The BC effect was measured directly using homemade equipment, where the temperature variation of the specimens was measured after a compression and decompression procedure. By these measurements was obtained a maximum temperature variation of ~14 K at a pressure of 218 MPa at 293 K for corn oil and ~13.7 K at a pressure of 218 MPa at 303 K for coconut oil, presenting values comparable to that observed in the literartura. The two investigated oils have the potential to be applied in cooling systems using the BC effect principle.

The Author thank the graduated program in Mechanical Engineer/ State University of Maringa (PEM/UEM)This work was supported by CNPq

Giant Barocaloric Effects in Natural Rubber Cup Lumpand Technical Specified Rubber TSR-10

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Natural rubber produced from ***Hevea brasiliensis*** has played a crucial role in the development of Brazil''s economy since the early 19th century. Nowadays, Brazil remains a significant producer of natural rubber, with the majority of its output being used for domestic consumption. The country's importance in the global rubber market has also been revived by the growing demand for sustainable and eco-friendly products. In parallel, solid-state refrigeration based on barocaloric effect has garnered global attention as a promising eco-friendly substitute for conventional vapor-compression refrigeration. In our work, we demonstrate for the first time giant and reversible barocaloric effects in natural rubber cup lump and in technical specified rubber TSR-10, both at near room temperature and over a wide temperature range. Giant adiabatic temperature change and giant isothermal entropy change up to 14 K and 100 $J.kg^{-1}.K^{-1}$, respectively, were achieved for a maximum pressure change of 250 MPa. The results are comparable to the best barocaloric materials reported so far, encouraging new prospects for the application of more sustainable and renewable resources of solid refrigerants in the near future of solid-state cooling devices.

Glass nano-structuring with argon as etching gas

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A major issue for the installation of photovoltaic modules is dust accumulation, known as soling, due to environmental conditions. This is a major problem in places where water is scarce, like desertic areas, which at the same time, have a enormous potential for photovoltaic energy due to its high solar irradiance. Thus, self-cleaning modules, with hydrophobic surfaces, come up as a solution for an efficient use of the available resources. Materials wettability properties can be modified by changing the micro or nanostructure of their surface [1]. For this purpose, a metallic film of few nanometers is deposited on the glass, then, through annealing, the film undergoes a dewetting process forming islands which will be used as masks during glass etching. Finally, the glass surface is modified through reactive ion etching (RIE) and the metallic film is washed out, obtaining a transparent glass with a nanostructured surface. Glass nanostructuring is commonly performed with gases like CHF3 or C4F8. However, C4F8 is an environmental hazard andCHF3 a particularly potent greenhouse gas, so their use should be avoided if possible. Inthis work, we nanostructured a glass surface through RIE by using only Argon as etchinggas and a gold nanomask. As a result, we grew amorphous nanostructures on the glass surface whose shapes and size depended on the initial thickness of the gold film, when keeping annealing temperature and RIE RF power as constant parameters. Using gold filmsof 5 nm and 20 nm resulted in nanostructures of 54±10 nm and 223±35 nm, respectively. Glass nanostructuring changed the wetting contact angle of the surface, from 35° (flat glass) to 50±5° and 67±5°, while optical transmittance decreased from 88% to 87%.

Acknoledgments:
ANID-Fondecyt 1210490; ANID-Fondap 15110019 and ANID-Millenium Science Initiative Program NNBP NCN2021_021.

References:

[1] Ruffino et al, Phys. Status Solidi Appl. Mater. Sci. 212, 1662 (2015)

Heterostructured $BiVO_4/V_2O_5$ semiconductors as photoanode for aqueous zinc-iodine batteries

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The development of chemical mechanisms towards storing energy has been increasingly a topic of notable relevance for our society. Redox batteries are interesting alternatives for storing electricity [1]. More specifically, zinc-iodine aqueous redox batteries (ZIRB) are interesting in several aspects, including low cost, high chemical stability, safety on handling and friendly environmental characteristics. On charging, $I^-$is oxidized and promotes the reduction of $Zn(OH)_4^{2-}$; the reverse process occurs during its discharge [2]. In this work, the $BiVO_4/V_2O_5$heterojunction was used as a photoanode to accompany the process of photoassisted battery charging. Under illumination, the stimulated photogenerated charges, at the electrode/electrolyte interface, promotes the oxidation of I^-, thus dropping the charge potential. The structural and morphological characterization of the synthesized materials in the laboratory were performed with different techniques, namely X-ray diffraction, transmission electron microscopy and scanning electron microscopy. The system efficiency was evaluated by voltammetry, chronopotentiometry and potentiostatic impedance. In order to simulate the sunlight spectrum, the photobattery was illuminated with an Xe light source. Charging the battery at a current density of 0.34 mA cm^{-2}, in dark conditions, required a potential of 2.43 V, whereas, under light, the potential for the same charge dropped by ~11.9%, reaching a specific load capacity of 500 mA h g^{-1}. The present results add to the technological development in the field of photoelectrochemistry to building water redox batteries, along with boosting further research and development on chemical storage of electricity generated from renewable energy sources.

Acknowledgments

We thank FAPEMIG, CNPq and CAPES for their financial support.

References:

[1] Q. ZHANG and J. LUAN, AngewandteChemie International Edition 59, 13180 (2020)

[2] M. DONG and Y. WANG, Nanotechnology 33, 265401 (2022)

Hydrogen storage in Ti-V-Nb-Cr alloy produced from commercial raw materials and scraps

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Hydrogen is a promising energy carrier [1]. Metal hydrides and multicomponent alloys are studied for hydrogen storage and designing materials with optimized properties. The Ti-V-Nb-Cr system has been extensively studied [2], but high-purity elements are often used. In this work, the (TiVNb)65Cr35 alloy was synthed by arc melting using scraps and commercially pure elements. Ti was added by using Ti-CP2 machining chips, commercially Nb from Brazilian Company of Metallurgy and Mining, and Cr from steel making industry were employed. Only high purity V was employed since no commercially pure V was available. Raw material and alloy were characterized by X-ray diffraction and scanning electron microscopy, and the hydrogen storage properties evaluated via SETARAM Sieverts-type apparatus. Differently from the same alloy produced from high purity elements that presented body centered cubic structure, the (TiVNb)65Cr35 produced here solidified with single C15-type phase structure (a = 7.045Å). The alloy was able to absorb 0.6 H/M withoutany activation treatment. Therefore, it was shown that the use of less pure alloying elementsfor alloy production can affect both structure and, consequently, hydrogen storage properties of alloys with same nominal composition.

Acknowledgment:
This work was financed in part by the Serrapilheira Institute (grant number Serra-1709-17362) and in part by the Brazilian National Council for Scientific and Technological Development - CNPq (Grant number: 309467/2021-7 and 407906/2022-3). This study was also financed in part by the Coordenação de Aperfeiçoamento de Pessoal deNível Superior - Brasil (CAPES) - Finance Code 001, Federal University of Sao Carlos, Graduate Program in Materials Science and Engineering. References:

[1] A. Züttel, Materials for hydrogen storage, 2003.
[2] R.B. Strozi, D.R. Leiva, G. Zepon, W.J. Botta, J. Huot, Effects of the Chromium Content in (TiVNb)100−xCrx Body-Centered Cubic High Entropy

Hydrogen Storage Properties of Novel TiZrNbCrNiCoand TiZrCrMnFeCo Multicomponent Alloys

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"Multicomponent alloys" is a class of materials that can offer countless possibilities for new metal hydrides with tunable hydrogen storage (H_2S) properties. In these applications, the alloys are usually prepared by a simple mixture of five or more A and B elements (A: elements forming hydride, B: elements with low chemical affinity with hydrogen), in equiatomic or non-equiatomic fractions that range from 5 to 35 at% [1, 2]. In this study, we investigate the crystal structure and the hydrogen storage properties of two novel equiatomic multicomponent alloys: TiZrNbCrNiCo (AB-type alloy) and TiZrCrMnFeCo (AB_2-type alloy). These compositions were selected based on thermodynamic calculations that indicated a high tendency to form C14 laves phases, which is beneficial for storing hydrogen at room temperature. The alloys were obtained by arc melting under an inert argon atmosphere. Characterization techniques such as XRD with the Rietveld refinement method, SEM/EDS, and TEM were used to analyze the alloys' microstructures. The hydrogen storage performance will be evaluated using absorption/desorption kinetics and PCT isotherms. Preliminary results indicated that both alloys have a single C14-laves phase, in accordance with the thermodynamic calculations provided by the CALPHAD method. It is expected that both alloys show an interesting hydrogen storage capacity with good absorption/desorption kinetics. The H_2S results are expected to be distinct, due to the different fractions of A andB elements between the compositions.

Acknowledgements:

Brazilian Research Funding Agency FAPESP (Project No. 2022/01351-0)

Coordination of Superior Level Staff Improvement CAPES (001)

References:

[1] Floriano R, et al. IJHE, 45, 33759-33770 (2020)

[2] Andrade G, et al. IJHE, 48, 13555-13565 (2023)

Improvement of hydrogen storage properties of Mg/MgH_2 through the addition of intermetallic TiFe

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The hydrogen storage in solid state can be achieved safely and conveniently through various alternative materials. The Mg/MgH_2system is a strong candidate in solving hydrogen storage due to its high gravimetric (~7.66 wt.% H_2) and volumetric (~110 kg $H_2.m^{-3}$) capacities. However, the need for additives in Mg/MgH_2-based systems is necessary to accelerate the kinetics and reduce the H_2absorption/desorption temperatures. In a previous work [1], we evaluated the performance of Mg/MgH_2reactions with H_2, focusing on the effects of additives at temperatures below the equilibrium temperature of 283 ºC @ 1 atm, starting from room temperature. Traditional additives such as Nb_2O_5, Fe, TiFe, and TiAl evaluated in other studies at higher temperatures have shown to significantly affect the kinetics and conversion of Mg to MgH_2at lower temperatures. Among the tested additives, the best performance was observed by adding TiFe to Mg/MgH_2, which was attributed to the attraction of hydrogen to the high-energy interfaces between phases. Additionally, the TiFe intermetallic compound, composed of immiscible elements with Mg/MgH_2, proved to be stable during reactions with hydrogen. In this work, we review the results associated with the use of TiFe added to the Mg/MgH_2system based on the available literature and calculations of formation enthalpies at the interfaces between phases using the extended semi-empirical Miedema model [2]. Based on the possible role of interface energies, it is suggested that other Ti-based compounds may have a considerable effect on accelerating the kinetics and conversion of Mg to MgH_2, in addition to TiFe.

Acknowledgements:
Brazilian Agencies: CNPQ, CAPES, FAPESP, Instituto Serrapilheira.

References:

[1] Antiqueira, F. J. et al. International Journal of Hydrogen Energy, v. 47, n. 1, p. 470-489, 2022.
[2] Zhang, R. F. et al. Computer Physics Communications, v. 209, p. 58-69, 2016.

Influence of carboxylic groups on sulfonated melanin/PEDOT:PSS based supercapacitors

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Melanins have become promising materials for application in pseudocapacitive energy storage systems due to the relationship among the redox activities of their base monomer and the ability of different functionalizations to reversibly bind to cations [1]. In this work, we investigated how the concentration of carboxylic groups present in sulfonated melanins (SMel) affect the electrochemical performance of poly(3,4-ethylenedioxythiophene)-poly(styrene sulfonate) (PEDOT:PSS) as electrode in supercapacitors (SCs). The concentration of carboxylic groups of SMel was controlled by the synthesis process using temperature (SMel-T) or oxygen pressure (SMel-8P) for the monomers oxidation. The blend was deposited by drop casting on stainless steel and poly(vinyl alcohol)/phosphoric acid (PVA/H_3PO_4) gel electrolyte was used to assemble symmetrical solid-state SCs. Fourier transform infrared (FTIR) and X-ray excited photoelectron spectroscopy (XPS) results showed that the concentration of carboxylic groups increased for SMel-8P compared to the SMel, while decreasing for SMel-T. Cyclic voltammetry and galvanostatic charge/discharge results indicated an improvement in gravimetric capacitance after addition of melanins. We observed a dependence on the concentration of carboxylic groups, reaching values of 9.9 F $g^{-1}$and 0.8 Wh kg^{-1}after the SMel-8P addition. The best ion injection was attributed to the greater conductivity of the protons of the melanins with greater amounts of carboxylic groups, which helped in the doping of PEDOT:PSS, increasing its electrical conductivity. So, our results show that the addition of SMel leads to an improvement in the electrochemical properties of PEDOT:PSS and evidences the role of the carboxylic group in energy storage. The biocompatibility of the materials tested as electrodes allows their future application as wearable devices.

Acknowledgements:
FAPESP (2021/03379-7)

References:
[1] P.Kumar et al., Journal of Materials Chemistry C, 4, 9516 (2016)

Influence of synthesis methods on microstructure and electrical characteristics of $SrCoO_{3-\delta}$-$Ce_{0.9}Gd_{0.1}O_{2-\delta}$ composite cathode for solid oxide fuel cells

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Among renewable energy sources, the fuel cells, as electrochemical devices that transform chemical energy of certain fuels to electrical energy by redox reactions, have been disseminated as an important alternative to be considered [1]. The solid oxide fuel cells (SOFC) with intermediate temperatures (500 - 800 °C) of operation gained attention for electrical energy production because of their capacity to be fed with reformed fuels (e. g. ethanol) [2]. In this work, a cathodic material, the strontium cobaltite ($SrCoO_{3-\delta}$), was synthesized by the methods of copreciptation, combustion with urea, Pechini and gelatin. The synthesized powders were calcined at 1.000 °C for 4 hours and characterized by X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The synthesized powders were mechanically mixed with gadolinium-doped ceria ($Ce_{0.9}Gd_{0.1}O_{2-\delta}$- GDC) to produce the composite samples. The films were deposited on GDC substrates by the screen-printing method and, after, were sintered at 1.100 °C for 6 hours. Lastly, the cathodic films were characterized by Impedance Spectroscopy and SEM. With the chosen synthesis methods, it was verified their influence on microstructure and electrical characteristics. The synthesis method that produced the material with monophasic perovskite structure and lowest area specific resistance (ASR), of 0.0543 $\Omega\cdot cm^2$, was the Pechini method.

Acknowledgements:
To the UFPB, the Ceramic Materials Laboratory from DEMAT/UFPB and the LabFilm from DEER/UFPB for the all subsidies and conditions to research and analysis.

References:
[1] R.S.Amado, L.F.B.Malta, F.M.S.Garrido and M.E.Medeiros, Quimica Nova 30, 189-197 (2007)
[2] D.Saebea, Y.Patcharavorachot and A.Arpornwichanop, Journal of Power Sources 208, 120-130 (2012)

Investigation of ECAP prossessing on the microstructure of the Ti-29Nb-13Ta-4Nb alloy

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In the present work, we investigated the effect of Severe Plastic Deformation (SPD) process by Equal Channel Angular Pressing (ECAP) technique on the the Ti-29Nb-13Ta-4Nb alloy. As reported in the literature, the microstructure refinement by SPD can enhance the kinetics of hydrogen absorption and desorption in hydride-forming alloys for hydrogen storage [1]. The influence of the number of ECAP passes was analyzed up to 8 passes. Morphological and structural characterizations were carried out by confocal scanning laserMicroscopy (CSLM), scanning electron microscopy (SEM), and X-ray diffraction (XRD) to correlate the effects of processing parameters on the samples microstructures. The effect of ECAP in the samples was also assessed by nanoindentation tests. The morphological results obtained by SEM showed a reduction in grain as function of the number of ECAP passes. Nanohardness was increased as function of ECAP passes showing the higher value for 6 passes. Further characterization techniques were employed to fully understand the effect of ECAP on the microstructural evolution of the investigated alloy.

Acknowledgments:

The authors thanks the PRH/ANP/UFABC Program, the UFABC Multuser Central Facilities (CEM), and the funding agencies FAPESP and CNPQ.

References

[1] SCHLAPBACH, L. ; ZÜTTEL, A. Nature, v. 414, n. 6861, p. 353-358, nov. 2001.

Ion intercalation kinetics in Prussian Blue electrodesand analogues

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The present work investigates the intercalation kinetics of potassium ions in Prussian Blue electrodes and analogs (PB and PBAs), materials that have potential application as cathodes of rechargeable batteries due to their high theoretical capacity, low production cost, chemical stability, crystalline structure (a = ~10 Å) and are eco-friendly [1]. PB is a semiconductor that exhibits a three-dimensional framework structure with interstitial water molecules that can be replaced by alkali cations during charging. However, the diffusion of these cations through the PB structure is hindered by its low intrinsic electronic conductivity and poor ion diffusion coefficient, leading to slow intercalation kinetics [2]. The main objective of the study is to use electrochemical techniques to determine the diffusion coefficient and to describe the kinetics of intercalation of potassium ions in PB electrodes and PBAs grown by electrodeposition. The techniques employed are electrochemical impedance spectroscopy (EIS) and intermittent potentiostatic titration (PITT) [3]. Based on the existing literature, diffusion coefficients can vary between 10-5 to 10-15 cm²/s across different techniques, systems, and assumptions [4]. However, in our case, we have obtained diffusion coefficients in the range of 10-10 cm²/s for Prussian Blue electrodeposited at 0.3 VSCE in a electrolyte containing 1M KCl.The samples were also characterized electron microscopy, X-ray diffraction, and optical measurements. This study will contribute to the elucidation of the mechanisms involved in the charge/discharge processes in Prussian Blue electrodes which can contribute to their use in sustainable batteries that employ abundant non-toxic elements and aqueous electrolytes.

[1] Sha M. and Liu L., EEM Vol.3, 56-66 (2020)
[2] Wang Q. and Li J., InfoMat Vol.4, 22 (2022)
[3] Montella C., Electrochim Acta Vol. 51 3102-3111 (2006)
[4] Komayko A. and Arkharova N., J. Phys. Chem. Lett Vol. 13 3165-3172 (2022)

K+ intercalation in Prussian Blue

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Iron Hexacyanoferrate ($Fe_4[Fe(CN)_6]_3$), known as Prussian Blue (PB), is a material that presents physical properties that make it promising for application in battery electrodes [1]. PB is known for its chemical stability, non-toxicity, and low production cost. For having a crystalline structure with a lattice parameter around 10 Å, it easily accommodates ions in its crystalline interstice, an interesting strategy for applications in energy storage devices [2]. In this study, PB thin films were grown using the electrodeposition technique, and an electrochemical battery was simulated in order to study the impact of the insertion and extraction process of potassium ions (K^+) on the crystalline structure. Characterizations were performed on the samples before and after the battery tests: crystalline structure, morphology, chemical composition, and optical properties. The results reveal that the as-deposited films have a cubic morphology, face-centered cubic crystal structure (FCC) with a lattice parameter of 10.21 Å and preferential orientation [200], in addition to being a semiconductor with a direct gap. After the battery tests, a decrease in the lattice parameters of 0.79 % was observed by the insertion of ions, and a 1.89 % increase after the extraction of ions, without significant alteration in the crystalline structure, suggesting a process of intercalation of ions in the PB lattice. Characterizations made through Raman spectroscopy between the extraction and insertion tests indicate that the material is most stable under tests that end with the extraction process.

References:

[1] C. Zhang, Y. Xu, M. Zhou, et al, Adv. Funct. Mater. 27 (2017). doi:10.1002/adfm.201604307.

[2] H. Zhang, K. Xi, K. Jiang, X. Zhang, Z. Liu, et al, Chem. Commun. 55 (2019) 7910-7913. doi:10.1039/c9cc03156a.

Kröhnkite-type $Na_2Mn(SO_4)_2(H_2O)_2$ for use in thermochemical systems

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Thermochemical heat storage (THS) devices are consolidating as efficient solutions to energy problems [1]. In THS systems, the heat released (stored) is energy from the ruptureof chemical bonds in a reaction, i.e., endothermal heat. The storage features, as well as the storage density, correspond to the phase transition temperature of the PCM (phase-change material) [2]. In other words, a PCM material is a substance that absorbs and releases sufficient energy at phase transition to provide useful heat. In this work, the crystal Na2Mn(SO4)2(H2O)2 with a kröhnkite-type structure was explored for this purpose. The crystals were grown using the slow solvent evaporation method. The structural and thermal properties were investigated by X-ray diffraction (XRD), thermogravimetry (TG), differential thermal analysis (DTA), and differential exploratory calorimetry (DSC). XRD and Rietveld refinement analyses show that at room temperature, Na2Mn(SO4)2(H2O)2 crystallizes in a monoclinic system with space group P21/c. TG-DTA thermograms disclose thermal stability from 300 K to about 410 K, followed by structural changes due to transitions and phase transformations with increasing temperature. A dehydration latent heat of 169.38 kJ/mol was estimated from the DSC curve. The dehydration process takes place in 3 stages. XRD asa function of temperature (300-653 K), corroborates the transitions and phase transformations. Furthermore, a hygroscopicity test after thermal treatment at 583 K reveals partial rehydration after 24 h at ambient conditions. The results suggest that more studies should be carried out before assuming Na2Mn(SO4)2(H2O)2 as promising for applications in thermochemical energy storage devices.

Acknowledgments:

We thank the CAPES, CNPq, FAPEMA, and PPGCM-UFMA.

Reference

[1] Atul Sharma, V.V. Tyagi, C.R. Chen, D. Buddhi. Review on thermal energy storage with phase change materials and applications. Renewable and Sustainable Energy Reviews 13 (2009) 318-345.

Large compressive mechanocaloric effect on Natural Graphite/Thermoplastic polyurethane composites

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Solid-state refrigeration, based on i - caloric effects has been considered an economically viable and environmentally friendly alternative to replace conventional vapor cooling systems. Several types of materials have been classified as promising for application on solid-state refrigeration subject to the mechanocaloric effect. One of these potential materials are polymers which present good performance on elastocaloric, barocaloric, or twistocaloric effects. The investigations regarding polymers showed some of the desirable properties suggested by Lloveras and Tamarit [1] for their use as solid-state refrigerants. For example, these polymers could present giant ∆TS and ∆ST within a wide range of temperatures, under low to moderate pressures, besides good reversibility, good availability, and low cost. Despite showing a giant and supergiant barocaloric effect, polymers are challenging to be employed in mechanocaloric cooling devices due to their limitations, such as low density, low thermal diffusivity, and low thermal conductivity. Due to these unique characteristics, we investigated the compressive mechanocaloric effect of Natural Graphite/Thermoplastic polyurethane (NG/TPU) composites. They are promising candidates for caloric cooling applications, as they combine similar rubber properties with the processability of thermoplastics. The NG/TPU composites presented ∆TS = 8.59 K and ∆ST = 35.01 J kg−1 K−1, which correspond to a maximum reduction of 28% and 45% in the∆TS and ∆ST, respectively, when compared to pure TPU, but with an increase of 500% inthermal conductivity. Our findings combine a large mechanocaloric effect with the unique characteristics of TPUs and the enhanced thermal conductivity of its NG composite.

References:

[1] P. Lloveras and J-L. Tamarit, Advances and obstacles in pressure-driven solid-state cooling: A review of barocaloric materials. MRS Energy & Sustainability vol 8. 3-15 (2021).

Magnetic FeM (M = Ag, Co, Cu, and Ni) nanocrystals as electrocatalysts for hydrogen evolution reaction

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Over the past decade, the high demand for nonrenewable fossil fuels in diverse branches of society has occasioned excessive CO2 emission into the atmosphere, which provides an environmental imbalance that becomes a cause for global warming. Aiming the CO2 emission to decrease and improve air quality in diverse cities, many resources for clean energy production, such as hydrogen, sunlight, and wind, can be considered. Among these, green hydrogen gas is the most promising and can be produced from electrochemical water splitting via hydrogen evolution reaction (HER). Thus, here, iron-based magnetic nanocrystals (FeM@OAm, M = Ag, Co, Cu, and Ni; OAm = oleylamine) electrocatalysts were synthed via the oleylamine reduction of metal salts method. FeCo is arranged in a body-centered cubic unit cell, while FeNi, FeAg, and FeCu are in a face-centered cubic structure. All the samples have different morphologies with an average diameter of 6.4 ± 1.0 to 21.7 ± 5.1 nm, depending on the bimetallic composition. The samples have ferrimagnetic behavior with a low coercive field and high saturation magnetization values at room temperature. Among heterogeneous electrocatalysts synthed, FeCo nanocrystals show lower overpotential (−564 mV) in comparison to FeAg (−584 mV), FeNi (−666 mV) and FeCu (−591 mV). The Tafel plots showed that the hydrogen evolution reaction (HER) activity for electrocatalyst following a mixed Volmer-Heyrovsky reaction mechanism suggests that reaction Volmer is the determining step. In addition, all electrocatalysts showed stability in tests of continuous operations showing only low potential variation. The EIS study shows a system characterized by two time constants, which may relate to the kinetics of the HER related to the charge transfer kinetics and the hydrogen adsorption. Thus, these materials showed acceptable electrocatalytic performance toward HER in alkaline media with excellent physical stability and an abundance of active sites for HER.

Malt bagasse hydrochar decorated with Co nanoparticles for hydrogen fuel generation

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Hydrogen (H_2) gas has been presented as a sustainable and very promising alternative fuel considering its high potential energy (1.42×10^8 J Kg^{-1}). However, its widespread use as a fuel is restricted by the safety and feasibility problems associated with the current storage systems. One way to get around this problem is to develop catalytic hydrogen generation technologies based on solid hydrogen storages, such as sodium borohydride ($NaBH_4$) [1]. In this work, the hydrogen generation performance of new nanocatalysts based on Ni and Co supported on hydrochar (HC) synthesized from malt bagasse was evaluated. The HC was synthesized by hydrothermal route (150ºC; 14 h) [2] using previously dried and ground malt bagasse. A broad characterization of the HC material was conducted (SEM, FT-IR, CNHS and TG/DTG), and it was possible to verify the production of a HC that preserved oxygenated functional groups. Ni and Co nanoparticles were supported on HTC using a reductive chemical synthesis method. The hydrogen evolution reactions were carried out using $NaBH_4$aqueous solutions in the presence of different catalyst compositions. Thus, the nanoparticles were synthesized with different Ni-Co proportions: 0%:100%, 20%:80%, 40%:60%, 50%:50%, 60%:40%, 20%:80%, 100%:0%. From this comparative study, it was observed that the use of monometallic Co nanoparticles supported on HC was the best catalyst. Then, the effect of adding NaOH to the medium was also studied, with a significant improvement in yield and reaction kinetics being verified. In the optimal condition (i.e., Co/HC catalyst at 0.5 mol L^{-1}NaOH), it was possible to obtain an average rate for H_2production at room temperature of 0.09 mL H_2s^{-1}(33 mL of H_2).

Acknowledgements:
FAPEMIG (RED-00144-22), CNPq (405828/2022-5) and CAPES.

References:
[1] H. N. Abdelhamid, Int. J. Hydrog. Energy, v. 46, n. 1, p. 726-765, 2021.
[2] C. O. Fontoura, et al., J Anal Appl Pyrolysis, 168 (2022): 105775.

Metal-Organic Frameworks Derived Mn-doped Copper Oxide Composite as a New Electrocatalyst for Water Splitting

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The electrochemical water splitting is one of the most promising and renewable methods to produce clean H_2, however some shortcomings still persist, especially for the oxygen evolution reaction (OER) [1]. Therefore, new methodologies to obtain high performance electrocatalysts based on Earth-abundant metals has gained attention. Thus, this work reports the obtention of a Mn-doped copper oxide composite, named $CuO/Cu_2O/Mn10$, through calcination of Metal-Organic Frameworks (MOFs), applied as electrocatalyst for OER. The material was characterized by XRD, vibrational spectroscopy (FT-IR and Raman), confirming the crystalline phases and chemical composition. SEM-EDS shows a rice-like morphology and indicates the presence of Mn homogeneously distributed in the sample. Electrocatalytic performance of $CuO/Cu_2O/Mn10$ was investigated by linear scanning voltammetry (LSV), cyclic voltammetry and electrochemical impedance spectroscopy, in alkaline KOH solution (1.0 mol/L). The material shows an excellent overpotential of 355 mV (at J = 10 mA cm^{-2}) and Tafel slope of 78.4 mV dec^{-1}. These results are similar or superior to other CuO and Cu_2O-based materials in the literature [2,3].

Acknowledgments: This work was supported by CNPq, CAPES and FAPESQ.

References

1. HAMEED, A. et al. RSC Advances, 12, 45, 28954-28960, 2022.
2. OLOWOYO, J. O.; KRIEK, R. J. Journal of Physics and Chemistry of Solids, 169, 110868, 2022.
3. PERUMAL, S.; YANG, H.; SEO, J. Current Opinion in Electrochemistry, 38, 101238, 2023.

Microstructural characterization of SSZ-13 zeolites for membrane-based N2/CH4 gas separation

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Zeolite membranes have presented promising capacity for gas separation [1]. For instance, the SSZ-13 zeolite displays a 3D structure with pores of 0.38 nm, allowing separation of gas molecules of similar kinetic diameters such as N2 (0.36 nm) and CH4 (0.38 nm) [2]. In this study, the hydrothermal synthesis with TMAdaOH (organic structuring-directing agent) was analyzed for SSZ-13 synthesis based on two different Si precursors, TEOS [3] and colloidal silica (Ludox AS-40) [1]. The duration of the hydrothermal synthesis was also analyzed within 1-4 days. Assessment of the zeolite microstructural properties was done by X-ray diffraction, microscopy (SEM), infrared spectroscopy (FTIR), gas sorption and thermogravimetry. All samples were crystalline, with exception to the sample obtained with Ludox using 1 day hydrothermal synthesis. SEM analysis revealed that the samples produced by TEOS presented cubic geometry (1-3 μm) whereas the samples obtained with Ludox formed larger (~50 μm) and multifaceted particles. SSZ-13 particles had high specific surface areas (>600 m2/g) with microporous structure (pore size 0.755 nm - typical of the 3D pores on the SSZ-13 structure with 0.38 nm pore windows). The thermal behavior of the samples revealed a 24% mass loss at 700 °C. Finally, preliminary trials on membrane production were performed by hydrothermal synthesis on top of alpha-alumina tubes (both commercial and freeze-cast tubes). The influence of the Si precursor on the SSZ-13 structure and the duration of the hydrothermal synthesis allowed precise control of the SSZ-13 properties, revealing the most adequate conditions for membrane manufacture.

Acknowledgments:
The authors would like to acknowledge funding from Petronas.

[1] A. Aydani, A. Brunetti, H. Maghsoudi, G. Barbieri, Sep. Purif. Technol. 256 (2021) 117796.

[2] M.A. Carreon, J. Mater. Res. 33 (2018) 32-43.

[3] L. Zeng, Z. Yu, Z. Sun, Y. Han, Y. Xu, J. Wu, Z. Liang, Z. Wang, Microporous Mesoporous Mater. 293 (2020) 109789.

Microwave synthesis of zeolite LTL (K, Sr)

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Zeolite LTL has 12-membered-ring one-dimensional (1D) channels with an accessible diameter of 7.1 Å, which turns the intracrystalline diffusion resistance lower than those of small-pore zeolites. Due to its crystal anisotropy, zeolite LTL has attracted attention for applications such as heterogeneously catalyzed hydrogen production [1] and gas separation [2]. The conventional synthesis of zeolite LTL is under hydrothermal conditions using high-pressure vessels and heating with ovens. This work describes a conventional and microwave synthesis of zeolite LTL (K, Sr) and its characterization. The synthesis was performed with a gel composition of $2.3K2O$: $0.2SrO$: $Al2O3$: $9SiO2$: $160H2O$ at 150 °C [3]. XRD patterns showed that the sample prepared by conventional synthesis for 1 day was composed mostly of amorphous phase. Otherwise, sample prepared for 3 days displayed very sharp diffraction peaks assigned to the characteristic planes of zeolite LTL. The results of microwave synthesis indicated that 12 h was enough to produce crystalline zeolite LTL (K, Sr). FTIR spectra confirmed the zeolite structure with bands in frequencies characteristic of this material. Moreover, zeolite LTL (K, Sr) exhibited a cylinder morphology in SEM images. BET specific surface area (184.28 m2/g) and total pore volume (0.111 cm3/g) of zeolite from conventional synthesis are lower than those of microwave-synthesized sample (238.29 m2/g and 0.139 cm3/g). Therefore, microwave heating is a simple and effective technology that reduces the synthesis time of zeolite LTL (K, Sr).

Acknowledgments:

The authors are grateful for the financial support given by PETRONAS and the technical support provided by Centro de Microscopia da UFMG.

References:

[1] L. Gartzia-Rivero, et al. Part. Part. Syst. Charact, 31, 110 (2014).
[2] L. Wu, et al. Microporous Mesoporous Mater., 316, 110956 (2021).
[3] L.J. Garces, et al. Journal of Catalysis., 217, 107 (2003).

Microwave synthesis study of graphitic carbon nitridefor application in photoelectrocatalysis

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As with any material, the synthesis conditions affect the characteristics of the resulting system. Traditionally, (g-C3N4) is synthesized through the pyrolysis of urea [1] or melamine [2]. On the other hand, only a few papers discuss the hydrothermal treatment of the precursor before the calcination step [3]. The use of microwave-assisted techniques in synthesizing this material is not known to us. In this context, we developed a microwave-assisted hydrothermal synthesis route of (g-C3N4). The properties of the resulting material were compared with those observed for materials prepared by conventional routes, aiming to evaluate the existence of thermal and non-thermal effects. Our results show that using microwaves reduces the reaction time from 24h (under conventional heating) to 2h40 (under microwave heating). As a result, materials with similar morphologies, crystalline structures and photoelectrochemical responses are obtained. This result may be ascribed to the thermal effects of microwaves, which increase the heating rates.

Acknowledgements:

UFABC, FAPESP, CNPq, CAPES, LNNano, CEM, JSS, Pho2con.

References:

[1] WANG, Jingjing et al. Journal of Colloid and Interface Science, v. 610, p. 842-853, 2022.

[2] JAYARAMAN, Venkatesa AYAPPAN, Chinnadurai; MANI, Alagiri. Chemosphere, v. 287, p. 132055, 2022.

[3] SONG, Meiting et al. Journal of Colloid and Interface Science, v. 588, p. 357-368, 2021.

MODELLING GRAPHENE OXIDE AND PEDOT - BASED SYSTEMS

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Poly(3,4-ethylenedioxythiophene):polystyrene sulfonate, PEDOT:PSS, is a well-known polymer blend used in organic electronics due to its electrical conductivity, flexibility, and optical transparency. It is known that mixing this compound with Graphene Oxide (GO) in a defined proportion can decrease the electrical resistivity of the samples up to three orders of magnitude to be applied as transparent electrodes in organic photovoltaic devices [1]. This study presents a theoretical model using the Kirchhoff’ s Circuit approach for these systems to elucidate the reasons for having a minimum corresponding to 5% of PEDOT:PSS into GO aqueous dispersion. This Modified Nodal Analysis methodology simplifies the evaluation by using a square lattice representation [2]. After calculating the equivalent resistivity of the system, it reveals a minimum spot that fits well with the experimental data mentioned before. There are also studies showing that GO can enhance the sensitivity of flexible gas sensor devices for methanol detection using PEDOT:PSS as active layer, with a well-defined maximum spot [3]. The model was further applied to these systems, revealing the same agreement between theory and experimental data.

Acknowledgements:

The authors are grateful to CNPq and CAPES for fundings.

References:

[1] B. G. A. L. Borges et. al, Molecular orientation and femtosecond charge transfer dynamics in transparent and conductive electrodes based on graphene oxide and PEDOT:PSS composites, Phys. Chem. Chem. Phys., 2019, 21, 736-743.

[2] G. Strang and L. Freund, Introduction to applied mathematics, Journ. of App.Mech., 1986, 53, 2, 480.

[3] L. S. M., Alves Influence of Nanostructuring Sensors Based on Graphene Oxide and PEDOT:PSS for Methanol Detection, IEEE Sen. Jour., 2023, 23, 3, 1845-1853.

Nanostructures of Cuo and Cuo/Zno for Application in Energy Storage

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Electrochemical capacitors (supercapacitors) play a key role in the development of new technologies for energy storage applications [1]. This work aimed to investigate nanoparticles of pure copper oxide (CuO) and the addition of different proportions of zinc oxide (CuO/ZnO) in relation to possible properties suitable for electrodes for electrochemical capacitors. The nanostructures were synthed by combustion reaction, the first sample consisting of 100% CuO and the second 80% CuO with the addition of 20% ZnO as initial test parameters. The samples were characterized by X-ray diffraction (XRD), infrared spectroscopy (FTIR), thermogravimetric analysis (TGA). The results of the structural and thermal characterizations revealed the formation of the desired crystalline phases, with all well-defined diffraction peaks and high organization at long range, in addition to good thermal stability with a total mass loss of only 7%. The electrochemical performance of the synthed nanoparticles, from an electrode prepared for supercapacitor, was studied by means of cyclic voltammetry, electrochemical impedance spectroscopy, and constant current charge and discharge method, from which relevant results were obtained, indicating that the samples of the metallic oxides studied have promising properties for the desired application.

Acknowledgements:
The authors are grateful to PNPD/FAPESQ, beneficiary program Renewable Energy, for the financial support.

References:
[1] M.J. Mostazo-López, Y. Hatakeyama, S. Shiraishi, E. Morallón, D. Cazorla-Amorós. Chapter 14. Smart Supercapacitors. Fundamentals, Structures, and Applications, p. 321-358(2023).

Natural Graphite/Polydimethylsiloxane Rubber Composites as Promising Barocaloric Materials

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Solid-state cooling based on the *i*-caloric effect is being explored as a potential replacement for conventional vapor-compression refrigeration systems. Although recent research has shown that elastomers have potential as barocaloric materials for cooling applications nearroom temperature, their insulating nature presents a disadvantage that can compromise practical use, since higher heat transfer properties are typically desired for increased efficiency. To address this issue, we investigated the barocaloric effect in composites consisting of natural graphite and polydimethylsiloxane (PDMS) at different mass proportions. Our findings are promising for solid-state cooling based on barocaloriceffect, as the composites exhibit a combination of desirable properties. When natural graphite was added to PDMS, the adiabatic temperature change (ΔT_S) and isothermal entropy change (ΔS_T) of the resulting composites decreased in comparison to pure PDMS. For example, at 323 K for 390 MPa of pressure change, pure PDMS demonstrated ΔT_S = 30.6 K and ΔS_T = −130 J.kg^{-1}.K^{-1}, while PDMS with 40 wt.% of natural graphite showed ΔT_S = 20.2 K and ΔS_T = −68 J.kg^{-1}.K^{-1}. However, the thermal diffusivity of the composites increased from 1.18×10^{-7} to 7.3×10^{-7} m^2.s^{-1}, an equivalent increase of ~500%. The natural graphite/PDMS composites were found to be superior or comparable to other barocaloric materials in the literature in various developed figures of merit.

New mixed Tutton salts with composition $K_2Mn_{1-x}M^{II}(SO_4)_2(H_2O)_6$, M^{II}= Co^{2+}, Ni^{2+}, Cu^{2+}, and Zn^{2+} for use in thermochemical and optoelectronic devices

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Hydrated double salts have gained much notoriety in materials science and engineering, due to the structural, thermal, and optical properties that these crystals present, which are of commercial interest for the development of thermochemical energy storage systems and fluorescent devices [1, 2]. With this in mind, a mixed Tutton salts series were synthesized with composition $K_2Mn_{1-x}M^{II}(SO_4)_2(H_2O)_6$, M^{II}= Co^{2+}, Ni^{2+}, Cu^{2+}, and $Zn^{2+}$by the isothermal evaporation method after 14 days. The properties were characterized by X-ray diffraction (XRD), thermogravimetry (TG), differential thermal analysis (DTA), differential scanning calorimetry (DSC), and spectroscopic techniques. In addition, a computational study was conducted through Hirshfeld surfaces and crystal voids to identify intermolecular interactions and lattice-free spaces. The XRD results confirmed the unprecedented nature of the double salts, in which all samples crystallized in a monoclinic system (*$P2_1/c$*). In addition, the thermal analysis revealed that the crystals have thermal stability ranging from 76 to 96 °C. Complementarily, the values calculated from the DSC thermograms showed that all 4 Tutton salts synthesized here, present enthalpy values and energy density promising for use in thermochemical systems. Moreover, the spectroscopic data in the emission regime showed that all salts exhibit photoluminescence in the visible region, which makes these materials interesting for application in solid-state lighting. Besides, it is worth mentioning the low cost and efficiency in the analyzed physical properties of these single crystals.

Acknowledgements:
We would like to thank CAPES, CNPQ, and FAPEMA for their support in this research.

References:
[1] OUSALEH, Hanane Ait et al. Renewable Energy, v. 162, p. 447-457, 2020.
[2] MEHRALI, Mohammad et al. Chemical Engineering Journal, v. 405, p. 126624, 2021.

NI CATALYSTS DEPOSITED ON CARBON CLOTH PREPARED BY PULSED LASER DEPOSITION FOR ELECTRO-OXIDATION OF UREA

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We proposed to use carbon cloth (CC) as a support for nickel nanoparticles (NiNP/CC) prepared by Pulsed Laser Deposition (PLD) at three different deposition times (20 s, 40 s, and 60 s) as catalysts for urea electro-oxidation reaction (UER) in alkaline media. Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDS) were used to characterize the materials. The SEM analysis shows that they were deposited uniformly onto the CC surface and gradually agglomerated with the increase of deposition time. The electrochemical characterization by cyclic voltammetry (CV) in 1 mol L^{-1} KOH showed that the characteristics peaks of $Ni^{2+}[Ni(OH)_2]$ oxidation and Ni^{3+}(NiOOH) reduction were present in all three synthesized samples. Electrochemical Impedance Spectroscopy (EIS) depicted a less resistive material as the nickel deposition time increased. Regarding UER, the maximum current density from CV experiments (normalized by geometric surface area of the electrode - GSA) in NiNP/CC 60s catalyst was approximately 15% higher than that in NiNP/CC 40s and about 49% higher than that in NiNP/CC 20s. However, the results related to UER from CV experiments normalized by electroactive surface areas (ESA) showed the maximum current density obtained with NiNP/CC 20s catalysts was around 35.5% and 67.9% higher than that obtained with NiNP/CC 40s and NiNP/CC 60s catalysts, respectively. Chronoamperometry (CA) measurements demonstrated that all the three materials had suitable electrocatalytic stability towards UER after 30 min of experiment. The highest current density of CA was obtained using NiNP/CC 60s when normalized by GSA. However, NiNP/CC 20s was responsible to the highest current density from UER at the end of the experiment when normalized by ESA. EIS measurements proved that the charge transfer resistance significantly decreased for UER as the electrocatalytic activity of nickel nanoparticles increased.

Ni-Co bimetallic nanoparticles supported on Zn-C battery residual electrolytic paste as a sustainable catalyst for hydrogen evolution from NaBH$_4$

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Hydrogen has a high energy potential (1.42x108 J Kg^{-1}), being considered the future fuel. However, its storage process such as gas compression or liquefaction are still unfeasible due to its low density and boiling point [1]. Thus, hydrogen storage in solid state, such as NaBH$_4$, are considered promising alternatives. The release of H2 from these compounds must be catalyzed. Catalysts from e-waste consists in a sustainable alternative for recovering and adding value to these materials [2]. In this work, the electrolyte paste (EP) from used cells was used as support of nanocatalyst for H$_2$ evolution. Firstly, the material was dried, crushed, and sifted (< 150 mesh). The recovered material presented diffraction peaks that correspond to the patterns of ZnMn$_2$ O$_4$ (hetaerolite), graphite and Zn$_5$(OH)8Cl$_2$·H$_2$O (simonkolleite). By FTIR, bands were observed that were attributed to MO bonds M and MO (M = Zn, Mn), Co-O and Ni-OH. By Raman spectroscopy, characteristic graphite bands were observed at 1354, 1580 and 2697 cm^{-1}. Bimetallic nanoparticles of cobalt and nickel (2:8), Ni-Co NPs, were supported on this material by chemical reduction with NaBH4, with bands corresponding to the vibrational mode of Ni-O being observed at 518 cm^{-1} and of Co$_3$O$_4$ at 470, 512, 604 and 667 cm^{-1}. By High Resolution Transmission Electron Microscopy (HRTEM), graphite-like sheets with an interplanar distance of 0.34 nm were confirmed. The nanoparticles had a of 1.35 + 0.67 nm. The catalyst showed a good performance in the evolution of hydrogen through the alkaline hydrolysis of NaBH4, at room temperature, with efficiency close to 90% in the first reaction cycle with activation energy of 41.1 kJ mo^{l-1}. The Ni-Co NPs (2:8)/EP material showed significant efficiency for up to 16 catalytic reuse cycles and 11 catalytic cycles of durability and turnover frequency (TOF) from 315.9 mL H2 g catal^{-1} min^{-1}.

One-pot conversion of lignocellulosic biomass-derived compounds using hierarchical ZSM-5 zeolites as catalysts

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The use of lignocellulosic biomass as a renewable feedstock has been proposed as an alternative to decreasing the dependence on fossil fuels. Platform molecules such as 5-hydroxymethylfurfural (HMF) and furfural can be used to obtain several value-added products with applications as biofuels and biofuel additives through hydrogenation reactions [1]. Such reactions can be performed using a one-pot approach, assisted by a catalyst containing Lewis and Brønsted acid sites and secondary alcohol as a hydrogen source. Zeolite-based catalysts are widely used in the industry, but their usage in biomass valorization reactions is hindered due to diffusional limitations. Therefore, chemical treatments can be performed to form hierarchical zeolites, containing both micropores and mesopores, to increase the efficiency in the conversion of the reagents and the catalyst lifetime [2]. In that context, this work aims to evaluate the viability of using ZSM-5 hierarchical zeolites with different Si/Al ratios (23, 38, 48, 50) formed through top-down methods and its usage in valorization reactions for the production of alkyl levulinates using domino-reactions. A desilication treatment was performed, and the effects on the structure and acidity of the zeolites were accessed through XRD, XRF, FTIR-Py, and TPD-NH3. Catalytic studies and design of experiments (DOE) were also carried out. As a result, the alkali-treated Z23 zeolite presented the highest FUR conversion and selectivity to isopropyl levulinate (98 and 95%, respectively).

Acknowledgments: CAPES, CNPq, FAPERJ and to Laboratório de Reatores, Cinética e Catálise (RECAT-UFF)

[1] FILICIOTTO, L. ; PFAB, E. ; LUQUE, R. Role of Biomass in the Production of Chemicals. In: Biomass Valorization. Wiley, 2021. p. 1-21.

[2] MARDIANA, S. et al. Hierarchical zeolite for biomass conversion to biofuel: A review. Fuel, v. 309, n. July 2021, p. 122119, fev. 2022

On the Compressive Mechanocaloric Effect of Natural Graphite/Thermoplastic Polyurethane composites: A Fatigue Analysis

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Replacement of vapor cooling systems with less impactful technologies is one of the many forms to address climate change. Solid-state refrigeration is one of the promising solutions as it virtually eliminates the use of greenhouse gases by replacing them with solid refrigerants. Concerning barocaloric technology, the research focuses on discovering barocaloric materials that can be applied as solid-state refrigerants. One of these potential barocaloric materials are polymers that present giant values of ΔT_S and ΔS_T within a wide temperature range for elastomers like natural rubber (NR)[1], acetoxy silicone rubber (ASR) [2], and commercial thermoplastic elastomers [3]. Despite the expressive number of works reporting the giant barocaloric effects. To date, no results of fatigue analysis have been reported in the literature considering materials with a compressive mechanocaloric effect (CME). In this work, we report the influence of the number of mechanocaloric cycles in the ΔT_S for the TPU and Natural Graphite/Thermoplastic polyurethane (NG/TPU) composites. The TPU and the composites with 20 and 40 wt% of NG/TPU were subjected to a rigorous fatigue test by cyclic compressive stress at 130 MPa (0.3 s^{-1}) in an experimental apparatus developed by the research grou at the end of the application of 10^5 cycles of confined compressive stress, no deterioration of the mechanocaloric compressive effect was observed.

References:

[1] E.O. Usuda and N.M. Bom et al., **Large barocaloric effects at low pressures in natural rubber**. Eur Polym J vol 92. 287-293 (2017).

[2] W. Imamura and E. O. Usuda et al., **Supergiant Barocaloric Effects in Acetoxy Silicone Rubber over a Wide Temperature Range: Great Potential for Solid-state Cooling**. Chinese Journal of Polymer Science vol. 38. 999-1005 (2020).

[3] N. Weerasekera and K.P.K Ajjarapu, **Barocaloric Properties of Thermoplastic Elastomers**. Front Energy Res vol10. 887006 - 10 (2022).

Oscillating magnetic effect in $BiFeO_3$ samples: apossible application in batteries.

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The development of electric cars led to the search for increasingly efficient batteries that bring less damage to the environment. In this context, it is evident that the study of new materials and effects to increase this efficiency is of fundamental importance. One of the materials that has characteristics that contribute to improving the quality of batteries is bismuth ferrite ($BiFeO_3$, or BFO), which is a perovskite that has been widely studied because it has characteristics suitable for various applications [1,2]. For example, its spontaneous magnetization can be controlled by a magnetic or electric field. Thus, in this work we carried out a detailed study of this ferrite using oscillating magnetic fields and observed that the BFO is sensitive to the frequency and shape of the magnetic fields. In fact, we observed a new effect through the transmission of electromagnetic signals on the perovskitesurface. The modulated magnetic ordering is important for the production of field sensorsbased on the oscillating magnetic effect, because when in the presence of magnetoelectriccoupling it is possible to detect magnetic fields through electrical output signals, which is important to evaluate the state and efficiency of the battery.

Acknowledgements:

This research was supported by the Brazilian National Council for Scientific and Technological Development (CNPq), Coordination for the Improvement of Higher Education Personnel (CAPES), Financier of Studies and Projects (FINEP) and Foundation for Support to Science and Technology of the State of Pernambuco (FACEPE).

References:

[1] N. Wang, X. Luo, L. Han et al. Structure, Performance, and Application of $BiFeO_3$ Nanomaterials. Nano-Micro Lett. 12, 81 (2020).

[2] L. Duraia, B. Moorthy, C. I. et al. Electrochemical properties of $BiFeO_3$ nanoparticles: Anode material for sodium-ion battery application Lignesh. Materials Science in Semiconductor Processing 68, 165-171 (2017).

Palladium nanoparticles immobilized on graphene derivatives for hydrogen production via catalytic dehydrogenation of formic acid

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Our group conducted a study in which palladium (Pd) nanoparticles (NPs) were immobilized on Reduced graphene oxide (RGO) functionalized with amine groups (Pd/RGO-NH2) using a simple wet reduction method. The presence of NH2 groups on the GO surface allowed the production of highly dispersed NPs with reduced s. This system exhibited exceptional catalytic performance for the mild decomposition of formic acid (FA), demonstrating total selectivity for the dehydrogenation route and complete suppression of carbon monoxide (CO) formation. In fact, we produced 78 mL of gas at 45°C in just 60 minutes, with an apparent activation energy value of 19.93 Kj.mol-1 and a hydrogen production rate equivalent to 65 mL.H2.min-1.g-1, which is comparable to the most effective heterogeneous Pd catalysts in the literature [1, 2, 3]. According to Han et al. (2021), the remarkable catalytic kinetics of our system may be attributed to the and high dispersion of the NPs on the support, as well as the contribution of the NH2 groups in the deprotonation of FA and the rapid formation of the formate ion (HCOO-). Moreover, recycling tests indicated that the catalytic activity remained stable until the fourth reaction cycle.

Acknowledgments:
To CAPES, FACEPE, and the SUPRAMMAT group.

References:
[1] HAN, J. et al. Journal of Colloid and Interface Science, v. 587, p. 736-742, 2021.
[2] HONG, C. B. et al. Inorganic Chemistry Frontiers, v. 6, n. 4, p. 975-981, 2019.
[3] KIM, Y. et al. Journal of Catalysis, v. 404, p. 324-333, 1 dez. 2021.

Photoanodes based on titanium dioxide and graphene oxide for application in hydrogen production

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Photoelectrochemical water splitting into H_2 and O_2 using semiconductors materials as photoelectrodes is a promising approach for obtaining hydrogen gas (H_2) [1]. TiO_2-based photoanodes have been widely investigated for this application due to their natural abundance, chemical stability, non-toxicity, high resistance to photocorrosion and low production cost. However, TiO_2 presents limitations such as low electrical conductivity, limited time in the excited state, and small diffusion distance. Incorporating graphene into TiO_2 increases the photoelectrochemical activity of the resulting material, attributed to the favorable band alignment of the two semiconductors and the properties of graphene that facilitate charge separation [2].

Here we report how the unique interactions of microwaves with the reactional media can produce TiO_2-graphene composites with enhanced properties. The method of synthesis used was adapted from previously reported conventional synthesis [2], with adaptations for microwave conditions. Also, ammonium sulfate was added as a capping agent to orientate the TiO_2 growth. Raman data show that it was possible to form anatase TiO_2 under all the synthesis conditions investigated. Additionally, it was possible to evaluate the degree of graphene reduction, given that reduced graphene oxide is more conductive. Adding ammonium sulfate to the reaction medium also increases the degree of graphene oxide reduction when the reaction is microwave-assisted but has no impact on conventional synthesis. These differences show that the unique effects of microwave irradiation allow one to obtain materials with characteristics distinct from those observed in materials produced by traditional routes.

Acknowledgements:
FAPESP, CNPq, CAPES, CEM-UFABC e PPG-CTQ-UFABC.

References:
[1] S. Kment et al., Chem. Soc. Rev., 46, 3716 (2017).
[2] L. Liu et al., ChemSusChem, 7, 618 (2014).

Photocatalytic fuel cells: fundamental concepts and applications

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The energy and environmental pollution crisis has become a recent worldwide concern. Photocatalytic fuel cells (PFC's) are energy converter devices that harvest solar energy using low-cost semiconductor electrodes to spontaneously convert the chemical energy of renewable fuels (or pollutants) and oxidants directly into electricity [1]. PFC's integrate photoelectrochemical and fuel cell technologies, having double benefits, i.e., to address environmental and energy problems. Since it has a simple engineering concept and uses only light as a driving force, low-cost PFC devices can be easily built to solve local environmental pollution or low-power energy-consuming problems [2]. This work presents essential definitions, working principles, and theoretical fundaments, including a hands-on tutorial on building devices and testing half-cell reactions and PFC's performance. In addition, some recent application cases will be presented and discussed in detail, focusing on critically debating strategies for boosting PFC's efficiency and stability.

Acknowledgments:

The author thanks CAPES, CNPq, and FUNDECT for the financial support.

References:

[1] B.D. Queiroz, J.A. Fernandes, C.A. Martins, H. Wender. J Environ Chem Eng. 10, 107611 (2022).

[2] K.-E. Guima, L.E. Gomes, J. Alves Fernandes, H. Wender, C.A. Martins. ACS Appl Mater Interfaces. 12, 54563 (2020).

Preparation and evaluation of P3HT:PC61BM photovoltaic devices with different contents of graphene oxide (GO) or reduced graphene oxide (rGO)as active layer

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Poly(3-hexylthiophene), known as P3HT, is a semiconductor homopolymer that is considered a reference material for research in the field of organic photovoltaic devices (OPV)[1]. The PC61BM is his standard electron acceptor. Graphene is a very famous carbon allotrope, mainly due to its excellent set of properties, such as excellent electrical conductivity, transparency, flexibility and mechanical residence. Current methods of obtaining graphene are still not efficient enough to produce material on a large scale while still presenting good quality and properties. Graphene has also been applied in the field of photovoltaic devices. However, there is a difficulty in standardizing and controlling the quality of the materials obtained, which makes it difficult to compare the results among those surveyed around the world and, finally, compromising the understanding of the impact of this nanomaterial on photovoltaic devices. The present work synthesized P3HT by the GRIM route, graphene oxide(GO) and reduced graphene oxide(rGO) by the Hummers method improved by the research group. Then, a ternary mixture of P3HT:PC61BM (1:1) with different levels of GO or rGO added (0.5%; 1.0%; 2.5% and 5.0%) were prepared. Then, solar devices were manufactured using these materials as an active layer and the main photovoltaic parameters and morphology were evaluated. The GO, although it has lower electrical conductivity than rGO, improved the energy conversion efficiency from 2.6±0.3% to 2.8±0.1% mainly becauseit has oxygenated groups that add better affinity with P3HT, reducing exciton recombination. Both GO and rGO favor a higher Jsc due to the light trapping phenomenon. Preliminary studies also suggest that the insertion of graphene tends to preserve the efficiency of the devices for longer periods of time.

Acknowledgements:
Thanks to CNPq, FAPERJ, CAPES and Fraunhofer IAP for their support.

References:

[1]TIENNE, L.G.P.; PAULA, T.P.; MARQUES, MF.V. Mater Sci Eng B, v. 293, p. 116505 (2023)

Raman, FTIR and density study of Nd^{3+} doped tellurite glasses

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Tellurite glasses have risen scientific and technologic interest for having wide optical transmission window (350-5000 nm), good vitreous stability (ΔT >100 °C), low phonon energy (600-850 cm^{-1}) when compared to basic silicate glasses $SiO_2$and high linear refractive index value (n ~ 2). The chosen system 65TeO2-15Li2O-20ZnO presented excellent results with promising properties in photonic application. Our work consists in the structural proprieties characterization of telluride glasses doped with $Nd_2O_3$for possible application in photonics. The samples were prepared using melting quenching technique, in ambient atmosphere. The obtained samples were characterized by the X-ray diffraction (XRD), a Raman spectroscopy technique, a Fourier transform infrared (FTIR) and using the density by Archimedes principle. The XRD analysis showed amorphous pattern without any spikes, which is typical for a vitreous sample. The Raman and FTIR techniques provided the information of a decrease in vibrational modes related to TeO_{3+1}unit. All other modes remain unchanged. These results means that the insertion of Nd_2O_3in composition does not alter the structural properties, meaning that we can consider this oxide as a dopant and not a network modifier. From density determination we observed that the oxygen packing density (OPD) increases up to 0,35% and then remains constant, this behavior complements the density results showing that the dopant increase the system density. The density increase is expected since we are adding a more dense material to the system.

Reverse Water Gas Shift Using Modified Titanate Nanotubes as Catalyst

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The search for ways to apply the Carbon Capture, Utilization, and Storage (CCUS) as strategy to CO_2mitigation has received relevant attention in the literature [1]. As $CO_2$is a thermodynamically stable molecule, their conversion in valuable chemical products is difficult. In this context, the reverse water gas shift (RWGS) can be used as an essential stage to the transformation of CO_2, which contribute to the mitigation of this gas. However, RWGS presents a bigger challenge that is the design of new catalyst that are capable to promote this conversion [2]. Titanate Nanotubes (TNT) modified with metals has been showing as a promisor catalyst to the CO_2conversion, presented high values of conversion and selectivity [3] and can be a good candidate to RWGS. In this context, the present work aimed to evaluate the efficiency of TNT modified with cooper (Cu), cobalt (Co), nickel (Ni) and iron (Fe) in the RWGS. From the better catalyst, the gas hourly space velocity (GHSV) was also evaluated. The results showed that Cu-TNT was the best catalyst, showing around 34% of CO_2conversion at 500 °C. In relation to the GHSV, the better result was obtained with 15000 $mL.h^{-1}g^{-1}$. Finally, a stability test was realized using the Cu-TNT and better GHSV. The result showed that catalyst was stable in 12 h of reaction leading the decrease less than 8% of CO_2conversion. These initial results indicate that Cu-TNT showed a promisor catalyst to RWGS reaction.

Acknowledgements

The authors would like to thank CAPES and CNPq for the financial support.

References:

[1] Boot-Handford et al., Energy Environ. Sci. 7, 130 (2014).
[2] Daza et al., Renewable Energy 176, 251 (2021).
[3] Monteiro et al., Fuel 253, 713 (2019).

Role of Rh2O3/CrxO3 cocatalyst in particulate Mo:SrTiO3 for green hydrogen production throughwater splitting.

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The alarming environmental problems are strongly related to the constant greenhouse gas emission, by the consumption of fossil fuel, as a mean energy source. This scenario highlights the necessity for new renewable and environmentally friendly energy sources, like green Hydrogen produced by water splitting, using sunlight and semiconductor photocatalysts [1]. A suitable candidate for this task is SrTiO3, due to its high photochemical stability in an aqueous environment and suitable energy band potentials for oxidation and reduction of the water molecule [1]. Despite its excellent characteristics, this semiconductor presents some drawbacks, like high recombination of photogenerated charges (electrons and holes) that demand modifications like the use of dopants and cocatalysts, to achieve high photocatalytic performance. In this work, was synthesized nanoparticulated molybdenum doped SrTiO3 by the molten salt method [2], and Rh2O3/CrxO3¬ cocatalyst was deposited into its surfacer by impregnation method. The physical properties of the photocatalyst were investigated by XRD, XPS, SEM, and PL. The photocatalytic activity of Mo:SrTiO3 was remarkably improved more than 700-fold for H2 evolution, after the deposition of Rh2O3/CrxO3¬. This behavior was attributed to the formation of a p - n junction of Mo:SrTiO3 (n-type) and the Rh2O3/CrxO3¬ (n-type), leadingto a high charge separation. The charge separation due to the p - n junction, was experimentally confirmed by photoluminescence measurements. This work highlights the excellent properties of Mo:SrTiO3/Rh2O3/CrxO3¬ as a photocatalyst for green H2 production through water splitting reaction.

Acknowledgment

FAPESP (2018/11059-0)

Refences

[1] R. V. Gonçalves, H. Wender, S. Khan, M.A. Melo, Nanoenergy, Springer Inter. Publishing, Cham, 2018: pp. 107-140.

[2] H.A. Centurion, L.G. Rabelo, I. Rodriguez-Gutierrez, M.M. Ferrer, J. Bettini, H. Wender, L.M. Rossi, F.L. Souza, R. V. Gonçalve ACS Appl. Energy Mater. 5 (2022) 12727.

Simulation of the Optical Properties of Multilayer Films Composed of Ag and ITO as an Artifice to Increase Electrical Efficiency in Crystalline Silicon Photovoltaic Cells

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Due to climate change, several measures to mitigate carbon dioxide emissions have been adopted, one of which is the use of clean and renewable energy sources to decarbonize the world's energy matrix. In Brazil, especially in the Northeast region, there are high rates of solar radiation throughout the year, therefore, the use of solar energy to generate electricity becomes viable and contributes to global goals. Although photovoltaic cell technology has developed a lot in recent years, there are still problems to be solved, such as the influence of cell operating temperature on electrical performance, which is inversely proportional. Thus, this work aims to propose, through mathematical simulation, an optical filter of solar radiation composed of silver (Ag) and indium tin oxide (ITO). Several multilayer configurations were analyzed and their optical properties simulated in order to obtain a filter with high transmissivity in the region of the solar spectrum in which the photoelectric effect occurs for generating electricity in crystalline silicon photovoltaic cells, as well as high reflectivity in the region of solar spectrum with few relevance for energy generation and that contributes positively to their heating, impairing electrical efficiency. Through the simulations, some configurations were compared, with different amounts of layers and thicknesses, highlighting one of 5 alternating layers of ITO and Ag with 85% of average spectral transmissivity from 400 nm to 900 nm and 80% of average spectral reflectivity of 1100 nm to 2500 nm, pointing out that the filter with the proposed materials has promising optical properties for spectral beam splitting applications in order to increase the electrical performance of photovoltaic cells exposed to high rates of solar radiation.

Acknowledgements:
UFPB, CNPq, FAPESQPB.

Spectroscopy and morphological characterization of semi-IPN conductive polymeric hydrogels for hybrid supercapacitors application

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The search for new methods of energy accumulation and storage is an important topic of research today [1]. In this regard, supercapacitors (SCs) are emerging as an alternative to conventional devices due to their performance and inherent advantages. Conductive polymer hydrogels (CPHs) are a new class of polymeric materials that combine the benefits of organic conductors and hydrogels. They exhibit remarkable synergistic properties aiming the expansion behavior and flexibility of hydrogels with the electroactivity of conductive polymers [2]. CPHs are also being used in the development of new energy storage devices, fuel cells, humidity-sensitive capacitors, photosensitive cells, and new SCs. The objective of this work was to perform spectroscopic and morphological analyses of CPHs composed of PAAm/CMC/PAni. It has been shown that CPHs, obtained from free radical polymerization [3] followed by interfacial polymerization using ammonium persulfate as an initiator, demonstrated good reproducibility and conductivity values on the order of 10-2 S/cm. The spectroscopic characterization showed characteristic bands of doped PAni, confirming its incorporation into the composites. The bands referring to polarons showed greater intensity, justifying the high conductivity of these composites. The morphological analyses showed PAni agglomerates filling the pores in the matrix, mainly closer to the surface of the hydrogels. The results also confirmed the formation of dendritic microcrystals attributed to the fractal growth of PAni. Finally, we can highlight that the composites presented good stability, reproducibility, and electrical performance, indicating their potential application as SCs.

Acknowledgments: UNESP, FAPESP, CAPES, CNPq, and FINEP.

References:
[1] K. Kyunghwa, et al, Applied Sciences., 9., 1547 (2019)
[1] H. Xinting, et al, Journal of Materials Chemistry A., 8., 23059 (2020)
[3] F. C. Tanaka, et al, Journal of Polymer and the Environment., 29., 3389 (2021)

SPINEL FERRITE MFe_2O_4 (M= Co, Ni, Zn) NANOPARTICLES OBTAINED BY HYDROTHERMAL ROUTE FOR OXYGEN EVOLUTION REACTION

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Transition metal oxides have gained prominence due to their optical, magnetic, and electrolytic properties. In this work, MFe_2O_4(M = Co, Ni, Zn) ferrite nanoparticles (NPs) with spinel structure were synthesized, characterized, and successfully employed for water splitting as electrocatalysts for both the hydrogen and oxygen evolution reactions (HER and OER). The NPs were synthesized by the hydrothermal method. Stoichiometrically calculated amounts of metal sources were dissolved in distilled water and transferred to a Teflon vial under stirring. After stirring, the vial containing the homogenized solution was placed in an autoclave, sealed, and heated at 180° C for 3 hours. All samples were washed three times with water and alcohol and dried at 80°C for 12 hours. The X-ray diffraction (XRD) pattern showed characteristic peaks which confirmed the NPs spinel ferrite crystal structure. Absorption bands were observed within the 560-450 cm^{-1}range of the infrared spectra, corresponding to vibrations of M-O bonds at tetrahedral sites and Fe-O bonds at octahedral sites. Furthermore, the optical behavior of the NPs showed a single absorption band in the visible region of the electromagnetic spectrum. While all NPs showed considerable HER and OER electroactivity, $CoFe_2O_4$presented the best performance for both reactions.

Structural properties of Cr_2O_3-doped tellurite glasses

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Tellurite glasses have attracted particular attention due to their properties, such as high thermal stability (~ΔT = 100 ℃) [1], low melting temperature (~700 ℃) [2], wide transmission range (~0.35 - 5 μm), low phonon energy compared to silicate glasses (~600 -850 cm-1), high linear refractive index value (~2) and high solubility for rare earth ions. These properties make this family of glasses a good candidate for optical and electronic devices. An excellent application that demonstrates the efficiency of optical glasses in photonics is the use of optical fibres, which are becoming an increasingly important part ofpeople's lives. The transition metals are widely used to modify glass structures due to itshigh radial distribution, by consequence, generating extremely sensitive responses to changes in interactions, [3] transition metals influence the behaviour of the optical properties by generating absorption bands in the near infrared region, inducing excited state absorption. Among the many transition metals, we highlight the chromium (Cr_2O_3) when used in high doping concentrations, it influences in the optical structural properties ofthe glasses. In this work, we characterise the system TeO_2-Li_2O-ZnO doped with different concentrations of Cr_2O_3: 0.1; 0.25; 0.50; 1.0, and 2.0 in mol%. The samples were prepared by the melting quenching technique. The structural properties were obtained using X-ray diffraction (XRD), Raman spectroscopy, Fourier transform infrared (FTIR) and density by Archimedes method techniques. The XRD technique was used to verify the amorphous stateof the synthetic materials. By Raman and FTIR techniques we observed that there is anincrease in TeO_3 modes as the Cr_2O_3 content increases. This behaviour indicates that Cr_2O_3 is acts as a network modifier from 740 cm-1. The oxygen packing density (OPD) obtainedfrom density values shows an increase in the packag of the glasses network as a consequence of these results.

Study of environmentally friendly routes to produce MXenes

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MXenes are 2D compounds that hold enormous potential for several technological applications, particularly in energy conversion and storage [1]. Despite their potential uses, producing these materials with high quality, i.e. contamination-free and defect-free lamellas efficiently exfoliated and delaminated, is a challenging task. Moreover, it is essential to develop environmentally friendly routes of exfoliation and delamination of the precursor MAX phase to obtain MXenes with adequate properties since the classical routes involve the use of concentrated fluoridric acid solutions. In this study, we investigated new synthesis protocols for producing the Nb2CTx MXene from the Nb2SnC MAX phase. The current literature suggests that obtaining a Sn-free MXene is challenging. To address this issue, we developed a new synthesis protocol based on the optimized routes designed for the Ti3AlC2 MAX phase. XRD, SEM- EDS, XPS, and TEM analysis indicated that Sn-free Nb2CTx MXene was obtained with good structural quality and chemical composition. These findings are significant for the advancement of high-quality MXene synthesis and may positively impact various application areas.

Acknowledgements:
The authors acknowledge the financial support from FAPESP (Grants # 19/22183-6; 20/15632-6; and 22/13307-6) and CNPq (Grant # 310984/2020-3). The Multiuser Central Facilities from Federal University of ABC (CEM-UFABC) is also acknowledged for facilities access.

References:
[1] R.M. Ronchi, J.T. Arantes, S.F. Santos, Ceramics International 45, p. 18167 (2019).

Study of environmentally friendly routes to produce MXenes and their uses in Supercapacitors

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MXenes are 2D compounds that have great potential for a variety of applications, including energy conversion and storage. However, their production with high-quality is challenging. The development of environmentally friendly delamination and exfoliation routes from the precursor MAX phase is crucial to minimize the impact of its production. Moreover, these synthesis protocols must be optimized for obtaining MXenes with the desired properties. This study focuses on the development of environmentally friendly routes to produce MXene with high structural quality and chemical composition for use in supercapacitors. New synthesis protocols were investigated for the production of Nb2CTx MXene from the MAX phase Nb2SnC, based on the optimized routes designed for the Ti3AlC2 MAX phase. The produced MXenes were analyzed by cyclic voltammetry and charge-discharge test, demonstrating potential for use in high-performance supercapacitors due to their unique properties of high electrical conductivity, high charge storage capacity, and high electrochemical stability. Thus, the development of environmentally friendly routes to produce high-quality MXene is a significant contribution to the advancement of MXene synthesis and the creation of more efficient and sustainable energy storage technologies. This study has a positive impact on research for new solutions for energy storage and environmental protection.

Acknowledgements:
Authors acknowledge the financial support from the FAPESP (grants # Grants # 19/22183-6; # 2022/13307-6) and CNPq (Grant # 310984/2020-3). The Multiuser Central Facilities of the Federal University of ABC (CEM-UFABC) is also acknowledged for facilities access.

STUDY OF MECHANICAL MILLED CARBON NANOTUBES USED AS CATALYST FOR ENERGYSTORAGE

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The development of hydrogen storage methods is one of the important areas of research in the field of hydrogen energy, while metal hydrides combined with nanocatalysts are the preferred candidates for this. Among these catalysts, multi-walled carbon nanotubes (MWCNTs) have been widely studied with the aim of increasing the hydrogen storage capacity and mainly improving the hydrogenation kinetics. MWCNTs synthed by traditional methods have high aspect ratio (length/diameter), with a diameter in the order of magnitude of a few nanometers and length in the order of magnitude of micrometers, limiting their applicability in many situations. Mechanical milling is an interesting option to modify the morphology of this material, being able to generate short and open nanotubes. However, little is known about the behavior of this material when comminuted into finer particles and how the mechanical comminution process interferes with its morphology/properties. The objective of this work is to study the morphological and structural effects generated by mechanical milling in MWCNT. For this, the material was comminuted for different times (20 min, 1h, 2h, 6h, 12h and 24h) under mechanical milling and the samples were characterized by different techniques, such as SEM, RAMAN, XRD, EDS. At the end, we want to evaluate the appearance of defects and new carbonaceous structures, in addition to the change in surface area. As the mechanical milling time increased, SEM identified that after 6 hours it was no longer possible to identify the tubular morphology of the MWCNTs. The use of EDS allowed observing a decrease in the carbon content present in the samples due to the appearance of new elements, such as iron. Raman showed that the structure of MWCNTs was completely modified. From the XRD, it was possible to observe that the peaks widen with the increase of the milling time, indicating the amorphization of the material.

Study vibrational and thermogravimetric of $NH_4KCo(SO_4)_2(H_2O)_6$ mixed Tutton salt for thermochemical energy storage systems

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Investigations of $X_2Y(SO_4)_2(H_2O)_6$ double-sulfated salts as thermochemical energy storage systems, where X(Y) is a monovalent(divalent) cation, have recently been reported [1]. In our first research on the $NH_4KCo(SO_4)_2(H_2O)_6$ mixed Tutton (named AKCoSH), the synthesis and a comparative structural study with its precursor salts were considered [2]. This work presents and discusses the vibrational property and the dehydration process of AKCoSH, which are important to phase change materials characterization aiming at the application in energy storage. Vibration modes analyses using FTIR and Raman spectra show contributions from the $[Co(H_2O)_6]^{2+}$ octahedral complex, $[SO_4]^{2-}$ tetrahedron, NH_4^+ cations, and coordination H_2O molecules. The assignments are based on those reported for the precursor Tuttons. The TG-DTA thermogram reveals that the AKCoSH is thermally stable from 300 up to 345 K. The first mass loss event is accompanied by an intense endothermal peak ascribed to salt dehydration. Complete rehydration was observed after 24 h. These results suggest the thermochemical application range using the AKCoSH. Three other thermal events (two Endo and one Exo between 495 and 600 K) in the DTA curve are observed; both are associated with mass loss stages. The attributions comprise the decomposition of an anhydrous compound and structural reorganizations. A crystalline phase transformation is supposed to arise due to the exothermal process around 600 K.

Acknowledgments: We thank the CAPES, CNPq, FAPEMA, and PPGCM-UFMA.

References: [1] Ousaleh, H. A. et al., Advanced experimental investigation of double hydrated salts and their composite for improved cycling stability and metal compatibility forlong-term heat storage technologies, Renew. Energy. 2020.

[2] Lopes, J.B.O. et al., Growth, structural and thermal properties of ((NH4)x(K1-x))2Co(SO4)2·(H2O)6(X = 0.0, 0.5, and 1.0) Totton salts In: XX Brazilian MRS meeting, 20, 2022, Foz do Iguaçu. Anais eletrônicos. p. 817.

Sunlight-driven water splitting using Cr-doped $SrTiO_3$ photocatalyst

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The development of products and technology is closely tied to the production of environmentally-friendly fuels, and one such strategy that has been employed is the use of photocatalysis to generate hydrogen from water. [1]. SrTiO3 has been widely studied as a semiconductor facilitator of photocatalysis reactions using sunlight, however, it has a band gap of 3.2 eV which causes it to absorb light only in the ultraviolet range (100-400 nm) meaning 4 - 5% of the solar spectrum [2]. Doping with transition metals such as Cr3+ increases light absorption in the visible range [3] and brings us promising results in the generation of H2. For this purpose, we have produced SrTiO3 semiconductors doped with Cr3+, and later, making use of the segregation effect, the material is decorated with Rh CrOx particles. The UV-vis spectroscopic analysis shows that RhCr/SrTiO3:Cr3+ has higher absorbance than the pure and Cr3+ doped material in the 400-700 nm range. The newly synthesized catalyst was able to produce 500 times more H2 (˜500 ·Mols after 5 hours of reaction) than the material only doped with Cr3+. The artificial photosynthesis reactions are conducted using a Xe lamp coupled with a 1.5 G AM filter in order to simulate the solar light.

Agradecimentos: RCGI, CNPq, FAPESP.

References
[1] Kanwal, Fariha; Torriero, Angel AJ. Energies, v. 15, n. 20, p. 7783, (2022)
[2] S. Ahuja, T.R.N. Kutty, J. Photochem. Photobiol., A Chem. 97, p.99, (1996)[3] C.H. Chang, Y.H. Shen, Mater. Lett. 60, p. 129, (2006)

Surface Active Ionic Liquids as Promising Materials for CO_2 Capture

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Carbon capture, utilization, and storage (CCUS) has been widely considered as a relevant strategy to reduce CO_2emissions [1]. For such application, ionic liquids (ILs) are known as promising materials for CO_2capture, and are less corrosive and volatile when compared with traditional amine-solvent processes [2]. However, a problem associated with these materials is their high cost, enhancing the search for new cheaper ILs that could maintain this materials advantages over benchmark technologies. Surface-active ionic liquids (SAILs) constitute a particular class of ILs that contain long hydrophobic chains in the cation, anion, or both [3]. This type of structure can provide water miscibility as well as cost reduction when compared with traditional ILs, such as 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([bmim][NTf_2]), one of the most studied ILs for CO_2capture. However, there are only a few studies about CO_2capture using SAILs. In this context, the present study aims the development of a series of SAILs using [TBA^+] or [$bmim^+$] as cations, and surfactant anions, and to compare their CO_2adsorption with [bmim][NTf_2]. The experiments showed that at 50 bar of pressure and 50 °C of temperature, the combinationof TBA as cation and lauryl ether sulfate (SLES) as anion resulted in adsorption values of101 gCO_2/kg IL, better results in comparison to the more expensive similar SAIL [bmim][SLES] (61 gCO_2/kg IL). In addition, these results are similar to [bmim][NTf_2] CO_2sorption performance (111 gCO_2/kg IL) allowing to conclude that the use of SAILs for CO_2capturing are promising and potentially viable.

Acknowledgements

The authors would like to thank Petrobras for the financial support.

References

[1] Jiang et al., Chem Eng J 136767 (2022);

[2] Elmobarak et al., Fuel 128102 (2023);

[3] Vieira et al., Catal Lett 148, 108 (2018).

Surface composition effects on the nickel-based electrocatalysts toward hydrogen evolution reaction

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Hydrogen has great potential as a primary energy vector for economic decarbonization because it provides through renewable energy sources[1]. So far, nickel-based efficient electrocatalysts for hydrogen production remain non-highly active towards the hydrogen evolution reaction. Here we investigate the surface composition effects of the dispersed Ru atoms on Ni electrocatalysts towards the rate of hydrogen evolution reaction in alkaline media. Deposited Ru atoms interact on nickel sites, leading to enhanced catalytic properties and, consequently, increase the adsorbed-hydrogen surface coupling reaction (Volmer step)[2] when compared to bare Ni catalysts. Although promised effects, the specific activity drops upon the limit concentration due to the formation of larger Ru particles on Ni surface.

Acknowledgements:
CAPES, ANEEL (PD-0394-2134/2021), PPGIES, PRPPG/UNILA, Fundação Araucária (nº PBA 2022011000128).

References:
[1]K.S Shiva and L. Hankwon, Energy Reports, v. 8, p.13793-13813 (2022).
[2]O.Zuraya Angeles et al, Energies, v. 15, p. 1609-1644 (2022).

Syngonnium biochar as catalysts for furfural condensation reactions

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Energetic transition took place in global discussions highlighting the need to find renewable sources to produce biofuels. Lignocellulosic biomass might be an alternative to produce biofuel by a bunch of reactions. This process requires a set of reactions to produce these chemicals, including furfural condensation. This reaction is significant because it can enhance the furfural carbon chain, producing the trans-4-(2-furyl)-3-buten-2-one, applied mainly as fuel additive and precursor for biofuel synthesis. Nevertheless, this reaction needs an acid or basic catalyst, in that aspect, biochars are sustainable materials that might be applied for that purpose, since they obtained from biomass pyrolisis. In this work, we propose the application of syngonnium (Syngonium podophyllum) biochar as a catalyst for furfural condensation reaction.1 The biochar was synthesized by calcination in the air atmosphere of milled syngonnium at 350 °C for 2 hours.2 Several techniques characterized the biochar structure. Elemental analysis and FTIR analysis confirmed that biochar presents the expected organic structure, containing aromatic groups, C-O bonds, and carboxyl and phenolic groups. DRX and FRX analysis showed that some inorganic compounds are present in the structure as ashes, composed mainly of calcium oxide and carbonate. These compounds are relevant for catalysis since they confer basicity for this material, and this basicity value (2,02 mmol/g) was also quantified by titration.2 Further characterization and catalytic evaluation still need to be performed to evaluate this material''s performance. However, this material showed a considerable potential to be applied in condensation reactions since it has many basic sites in its structure. Acknowledgments: CAPES, FAPERJ, LAMAR-CAIPE, GCVB, UFF, ReCat.References: [1] He, J; Qiang, Q; Liu, Song, K; Zhou, X; Guo, J; Zhang, Li, C. Fuel, 306 (2021) [2] Certificate, E. B. Eur. Biochar Found, 1 (2012).

Syntheses of Hierarchical Zeolites based on Transition metal cations and their application on the Catalytic propane dehydrogenation

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Zeolitic framework in which the aluminum atom is replaced by transition metals with octahedral-pentahedral-tetrahedral coordination is known as OPT materials. Therefore OPT materials OPT is built up with a mixed crystallographic structure with tetrahedral silicon atoms and penta or octahedral coordinated transition metal atoms.[1]

Propylene is a basic start-point molecule for a large of important products productions, such as: Acetone, acrolein, polypropylene, and other industrial products. [2] One way of propylene production is through heterogeneous catalytic propane dehydrogenation [3]. In this study three novel OPT materials containing Cromium (Cr), Niobium (Nb) , Tin (Sn) and vanadium (V) were synthed using a set surfactant polymer as structure directing agents. The materials were characterized by X-Ray Diffraction (XRD); Fourier-Transform Infrared Spectroscopy (FT-IR); and Transmission Electronic Microscopy (TEM). The catalytic properties of these OPTs materials were tested in a typic fixed-bed quartz reactor at continuous flow. The catalysts were were studied in their as made and H2 reduced forms. The XRD patterns indicated the formation of pure and crystalline structure of the OPTs materials. The spectra of FT-IR the interaction between SDA and the polymeric chains of OPT materials. The SEM images showed the morphologic of theses crystals and TEM images indicate the formation of mesoporous. The catalytic results indicate all theses materials have a potential as heterogeneous catalysts to propane dehydrogenation. The H2 pre-treatment increase the propane selectivity.

Acknowledgment:

FAPESP, CNPq and CAPES have supported this work.

References

[1] J. Rocha. Reviews in Mineralogy and Geochemistry. 57 (2005) 173-201.

[2] S. Chen. Chemical Society Review. 2021, 50, 33153354

[3] Z. Lian. ACS Catal. 2018, 8, 5, 4694-4704

Synthesis and characterization of NiCo-BDC catalyst for hydrogen production by NaBH4 hydrolysis

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Hydrogen is a versatile and clean energy carrier, and its use can supply the world''s energy demand. However, hydrogen generation is still mainly based on fossil fuels [1]. A hydrogen storage material that generates hydrogen on demand is sodium borohydride (NaBH4), and its hydrolysis requires the presence of a catalyst. Among catalysts for hydrogen production, Metal-Organic Frameworks (MOFs) have attracted attention, due to their large surface area and active sites [2]. In this work, NiCo-BDC was synthetized by impregnating nickel(II) nitrate solution onto Co-BDC and used for dehydrogenation of NaBH4. The MOF was characterized by FTIR, PXRD, and SEM-EDS. NiCo-BDC exhibited a pure crystalline phase, high hydrogen generation rate (707.15 mLmin-1gcat-1, 46 °C) and good activation energy (54.57 kJ mol-1). NiCo-BDC also presented high stability, having catalytic activity after 10 cycling tests.

Acknowledgments:

The authors would like to thank CAPES (643078/2021-00), CAPES/PRINT (194800/2018-00) and CNPq (141076/2019-4) for the scholarship grants. And M.A.O gratefully acknowledges the support from Mayor of Gdańsk Daniel Gabriel Fahrenheit Scholarship Program for Students and International PhD Students.

References:

[1] International Energy Agency., Glob. Hydrog. Rev. 2022, (2022)

[2] B. S. Barros, et al., Chemistry Select, 3, 7459-7471 (2018)

Synthesis and characterization of $NiCo_2O_4$/g-C_3N_4 material at different temperatures for fabrication ofhigh-performance supercapacitors.

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The present work illustrates an one-step synthesis method of graphitic carbon nitride grafted nickel cobalt oxide using pyrolysis method. The as-prepared nanocomposites exhibit high specific surface area and excellent electrochemical performances for supercapacitor applications [1]. In this work $NiCo_2O_4$/g-$C_3N_4$was fabricated at temperatures of 350°C, 450°Cand 550°C. Each temperature creates a new material coated on nickel foam as base electrode in the construction of a high-performance supercapacitor. The three materials were characterized by FESEM, XRD and FTIR techniques. FESEM imaging confirmed thatthe materials have a nanoscale structure with some porosity and high surface area, allowingions to move between the electrode and the electrolyte, turning the reactions faster. The $NiCo_2O_4$/g-C_3N_4-350°C, $NiCo_2O_4$/g-C_3N_4-450°C and $NiCo_2O_4$/g-C_3N_4-550°C electrodes were characterized by electrochemical methods such as cyclic voltammetry, charge-discharge test and electrochemical impedance spectroscopy. The $NiCo_2O_4$/g-C_3N_4-350ºC, $NiCo_2O_4$/g-C_3N_4-450ºC and $NiCo_2O_4$/g-C_3N_4-550ºC electrodes showed specific capacitances of 268.71F/g, 86.04 F/g and 54.15 F/g respectively at a current of 0.5 A/g. The fabricated materials presented a higher performance, since it combines the advantages of all components: good conductivity, good electrochemical performance and pseudocapacitive properties [2].

Acknowledgment:
The authors would like to thank the CAPES, the CNPq, the FINEP and the FAPERJ.

References:
[1] B. Cui, H. Lin, J. Li, X. Li, J. Yang, J. Tao, "Core-Ring Structured $NiCo_2O_4$ Nanoplatelets: Synthesis, Characterization, and Electrocatalytic Applications", Advanced Functional Materials, vol. 18, no. 9, pp. 1440-1447, 2008.
[2] Z. Wu, Y. Zhu, X. Ji, "$NiCo_2O_4$-based materials for electrochemical supercapacitors", Journal of Materials Chemistry A, vol. 2, no. 36, pp. 14759-14772, 2014.

Synthesis and fabrication of flexible nickel oxide/GCN-NS electrode for energy storage

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This work describes the synthesis of nickel oxides grafted on graphitic carbon nitride nanosheets through a simple synthesis. The as-prepared was coated in carbon cloth (substrate) as electrode for supercapacitors applications. Morphological characterization was performed by FESEM technique which confirmed the presence of NiO/GCN-NS in the sample composition and its high surface area. Electrochemical methods such as cyclic voltammetry and charge-discharge test were performed to demonstrates the good electrochemical performance of the material for supercapacitors applications. The electrode showed good specific capacitances of 50 mF/g at a current density of 2 mA/g. This study shows that carbon cloth electrodes coated with NiO/GCN-NS contribute to supercapacitor applications that require good energy storage and present relevant mechanical flexibility of the material.

Acknowledgements:

We would like to thank the financial supports by the Brazilian agencies such as CAPES (BEX5383/15-3), (PNPD-PhD scholarships) CNPq (304051/2014-4) and FAPERJ (E-26/110.087/2014, /213.577/2015 and /216.730/2015).

References:

[1] Babu, R.S. et al., 2018, Composites Part B: Engineering 143, 141-147 (2018).
[2] Santos, R. S., et al. Materials Letters 308, 131156 (2022).
[3] Samyn, Leandro Marques, et al. Micro and Nano Systems Letters 9.1, 1-10 (2021).

Synthesis, characterization and hydrothermal reduction of graphite oxide to obtain graphene sheets

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Graphene is an allotrope of carbon that has attracted widespread interest as it is a versatile material capable of behaving like a liquid crystal [1] and exhibiting remarkable transparency, mechanical, thermal and electrical conductivity [2]. This work aimed to obtain graphene sheets from the reduction of graphite oxide by hydrothermal route. The graphite oxide synthesis was achieved following the modified Hummers methodology [3] conducted under temperature control, below -10 °C, given the exothermic nature of the reduction reaction. To investigate the nematic phase, that is, the liquid crystals, a 100 W power reflector and a polarizing film were used, keeping it at a standard angle. For this purpose, a photographic camera and a polarizing lens were used. After sending the material was isolated. The presence of oxygenated organic functions in the reaction product was confirmed by infrared spectroscopy. In solution, proceed with the reduction of graphite oxide in a hydrothermal reactor, for 24 hours at a temperature of 170 °C. It was observed that the appearance of liquid crystal depends on the concentration, as the concentration of graphene sheets increases, more prominence gains this property. The presence of oxygenated groups in the material obtained after hydrothermal reaction was lower compared to the standard graphite oxide. Spectrum analysis also showed an increase in the band of C=C bonds, after the hydrothermal reaction, when compared to the standard graphite oxide band, indicating reduction of the material.

AcknowledgementsThe authors would like to thank the IFAL, campus Piranhas, for the scientific initiation scholarship.References:[1] Yadav, M. K.; Sangitra, S. N.; Panwar, N.; Rimza, T.; Pujala, R. K. and Kumar, P. Materials Chemistry and Physics, 287, 126305, 2022.[2] Lalire, T.; Otazaghine, B.; Taguet, A. and Longuet, C. FlatChem, 33, 100376, 2022.[3] Hummers, W. S. and Offeman, R. E. Journal of the American Chemical Society, 80, 1339, 1958.

Synthesis of sodium niobate perovskites for CO_2 conversion and water splitting aplications

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NaNbO3 has been shown to be a very promising catalyst for the photoelectrochemical disproportionation of water and CO2 photoconversion [1, 2]. However, in order to increase its catalytic efficiency, it is necessary to improve the lifetime of the photogenerated charges and the modulation of its visible light absorption capacity. These properties, in turn, are associated to morphological and structural control. This work aims to study the optimization of the synthesis of NaNbO3, through microwave-assisted hydrothermal routes [1, 2]. The efficiency was improved through in situ polymerization of graphitic carbon nitride (g-C3N4), as well as adding platinum nanoparticles as co-catalyst in order to reduce the recombination effects of photogenerated charges on the material. SEM images show that R1 and R2 gave rise to 1D wire-like species. Despite this, the MET images, suggest a distinct mechanism of growth of these structures. The growth mechanism apparently impacts the crystal structure of the resultant material, which show an orthorhombic phase with subtle changes, which may indicate the presence of spurious phases. These variations impact the way NaNbO3-R1 and R2 interact with g-C3N4 and Pt. Consequently, the photoelectrochemical response of the investigated materials.

Acknowledgments:
The authors gratefully acknowledge support from UFABC, PPG-CTQ (UFABC), CNPQ (130958/2021-2), CAPES (Finance code: 0001), FAPESP (grant # 2120/05958-4) and CEM-UFABC.

References:
[1] FERNANDES, D. et al.. Ceramics International, v. 47, n. 7, p. 10185-10188, 2021.
[2] LÓPEZ-JUÁREZ, R. et al.. Ceramics International, v. 40, n. 9 PART B, p. 14757-14764, 2014.

TEM characterization of MXenes produced by different synthesis protocols

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MXenes is a family of 2D layered compounds with outstanding properties derived from their layered structures, complex chemical bonding and surface terminal groups. Therefore, these materials have been considered highly promising for several technological applications, particularly those related to energy storage and conversion such as rechargeable batteries, fuel cells, electrolyzers, solar cells, supercapacitors, etc. To fully understand the structural features of such materials and the relationships with the properties of interest, advanced characterization techniques must be employed. In this work, MXene compounds ($Ti_3C_2T_x$and Nb_2CT_x) obtained by different synthesis routes were characterized by transmission and scanning-transmission electron microscopy. The characterization was performed using a transmission electron microscope, ThermoFisher Scientific model Talos F200X equipped with a 4 in-column SDD EDX detectors. The investigated samples were synthed employing different types of fluoride salts in the exfoliation process. Particularly for Nb_2CT_xMXene, obtained from the exfoliation and delamination of the Nb_2SnC MAX phase precursor, the synthesis protocol is critical since obtaining Sn-free MXenes and its characterization are challenging tasks. The microstructural features of the investigated MXenes were assessed particularly concerning the presence of crystallographic defects. STEM was also important to perform chemical mapping of the investigated samples. The interplay between the microscopy results and synthesis protocols are depicted allowing designing more appropriate routes to synthe and functionalize the investigated compounds.

The influence of PVP molecular weight in the synthesisof silver nanowires using a salt-assisted polyol method

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In the last decades, the demand for new materials to be used in electronic devices has increased. Among the materials that can be used in transparent conductive films (TCFs), are the silver nanowires (AgNWs). This class of material, when supported in glass or PET, e.g., has good conductivity, thermal and mechanical properties and also high transparency. In this work, the AgNWs were synthesized using the polyol method assisted by a salt and the influence of molecular weight of the capping agent polyvinylpyrrolidone (PVP) in the morphology and structure of Ag crystals was investigated. Different molecular weight of PVP (40 K, 120 K, 360 K and 1300 K MW) was used in the synthesis. The surface morphology of the products was observed by scanning electron microscopy and atomic force microscopy. The phase composition and structure were analyzed by X-ray diffraction (XRD) and the XRD patterns diffraction peaks correspond with the face-centered-cubic (fcc) crystalline phase of silver. Transmission electron microscopy was used to analyze the crystal structure, and UV-visible spectroscopy was used to follow the formation of Ag structures. The results indicate that PVP and its chain length have an important role in the formation of silver nanocrystals due to the adsorption and steric effects [1] and as high is the molecular weight of PVP, longer and thinner are the AgNWs as observed by other authors [2]. Different morphologies were obtained when changing the size of PVP chain. For smaller MW of PVP, particles and short wires were formed. Increasing the MW, nanorods and pyramidal particles were formed, and finally, forming nanowires when using the highest MW PVP of the experiments. Improving the aspect ratio of silver nanowires is important to construct TCFs, since the conductivity and transparency properties are improved when they are thinner and longer. Ref.: [1] Song, YJ., et. al, Nanoscale Res. Lett., 9, 17 (2014). [2] Yuyue Guo,et al, Inorg. Chem. Comm., 128, 108558 (2021).

Tungsten trioxide fibers with enhanced photoelectrocatalytic activity

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Tungsten trioxide (WO_3) with the band gap of 2.4-2.8 eV, is one of the most important semiconductors, especially for electronic devices, gas sensors, photocatalysis, and water splitters [1,2]. Compared with conventional methods, electrospinning is a versatile method and can produce continuous nanod oxide fibers [2]. In this work, we reported the exploration of photoanodes based on WO_3fibers on FTO through a simple electrospinning strategy. Firstly, 0.2 g of polyvinylpyrrolidone (PVP, Mw 1300000) was dissolved in 0.6 mL of ethanol and 2.0 mL of DMF with vigorously stirring for 2 h. Then, 1.0 g of $WCl_6$was added to the above solution and continued to be stirred for 1 h. The solution was transferred to a plastic syringe for the electrospinning process, under 15 kV, rate of 0.36 mL/h, 18 cm from the collector, with ambient temperature and relative humidity at 20 °C and 45 %, respectively. After, the samples were calcined at 500 °C for 1 hour (2 °C/min) [3]. Fiber morphology and structure were studied using X-ray diffraction and scanning electron microscope. The photocurrent activities of the as-prepared samples are evaluated by voltammetry and chronoamperometry measurements performed in Na_2SO_4solution (0.1 mol L^{-1}) under visible light and interfacial properties by electrochemical impedance spectroscopy. Experimental results demonstrate the WO_3nanofibers formation and photocurrent density can be up to 0.25 mA cm^{-2} at 1.0 V vs. Ag/AgCl. The Nyquist plots of electro-spun samples showed a smaller radius which indicates a lower resistance to charge transfer and higher transmission efficiency compared with WO_3particles. Therefore, current work might open up an avenue to develop novel and efficient photoelectrodes.

Acknowledgements:
To LADEMA, GMPC, LMEM, LARX and CNPq for funding (Proc. 142243/2019-1).

References:
[1] Yao, Y et al. Nanomaterials., n. 1809036 (2021)
[2] Wang, Y et al. Adv. Funct. Mater., n. 1809036 (2019)
[3] Song, K et al. ChemElectroChem, vol. 5, 322-327 (2018)

Unlocking the Potential of Deep Eutectic Solvent in ZnSe Nanoparticle Synthesis via Hot Injection Methodand Factorial Design

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Zinc Selenide (ZnSe) is a widely known semiconductor in the literature, but minor is known about their synthesis in Deep Eutectic Solvent (DES), which enables biological application. [1,2] The optical and electrical properties of ZnSe, are crucial to its application, depending on the size of its nanoparticles, which can be controlled through the hot injection method. In this study, ZnSe synthesis in DES was carried out using a 24 factorial design in duplicate. The parameters evaluated were the reaction temperature (50 ºC or 80°C), reaction time (1or 5 min), solvent (ethaline or glyceline), and atmosphere (ambient or inert). First, 10 mL ofDES was prepared, using choline chloride:ethylene glycol 1:2 (ethaline) or choline chloride:glycerin 1:2 (glyceline), followed by the addition of 1 mmol of zinc acetate. Then, the systemwas heated to the desired temperature. Reaching this temperature, a solution of reducedselenium was injected, and the time of reaction was initialized. The reduced selenium wasprepared in an inert atmosphere by adding 1 mmol of elemental selenium in a 5 mL aqueoussolution of 2 mmol sodium borohydride. By the end of the synthesis, each sample was separated into two aliquots, half of which were washed and redispersed in ethanol, and theother half, kept in DES without washing. XRD analysis in DES presented the formation ofZnSe, while the washed solutions presented selenium, also. The UV-Vis spectra showed absorption around 400 nm and a bandgap of 3.0 eV. The materials presented fluorescenceemission around 700 nm. From now, DLS, SAXS, and TEM characterizations will be performed to observe the particle size. Finally, it will be possible to evaluate the effects ofthe studied parameters and conclude how DES helps to stabilize the structure and size of the particles.

Acknowledgments:We thank FAPERGS, CNPq, and PRH-ANP for their financial support.

References:[1] Prog Mater Sci 83 (2016) 472-535.[2] J Mol Liq 316 (2020) 113753.

Use of Neem (Azadirachta indica) seed husks in natura and as briquette: A comparative energetic analysis

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Biomass is a renewable resource available on a large scale, which can be used as biofuel, and for its better application, characterization is necessary. Among the important parameters to evaluate the potential of a fuel is the Calorific Value, defined as the amount of energy released in the complete combustion. In this context, the work presents the comparative evaluation of the Higher Heating Value (HHV) of Neem seed husks (Azadirachta indica) in natura form and compacted form called briquette. Studies show that Neem biomass has a significative HHV, which represents a viable option as a renewable energy source, producing less greenhouse gas emissions compared with fossil fuels. To the calculation of the HHV was used an equation based on the results of the proximate analysis: HHV = 0.3536x(% FC) + 0.1559x(% VM) − 0.0078x(% A) [1], in which FC is the fixed carbon content, VM is the volatile matter content, and A is the ash content. The HHV results for Neem seeds and briquette were 17.82±0.29 MJ/kg and 17.60±0.17 MJ/kg, respectively. The briquette production from Neem seeds did not show significant variation in the HHV. However, the briquette facilitates the transport and storage of biomass for further use. Another possibility to increase the HHV is to prepare blends using different biomasses. Finally, the application of Neem seed husk did not change the properties during the briquette production process, being a favorable characteristic of this biomass.

Acknowledgements:
This work was supported by CNPq (308280/2017-2, 313647/2020-8); FUNCAP (PS1-00186-00255.01.00/21); FINEP; and CAPES (Finance Code 001).

REFERENCES

[1] Parikh, J; Channiwala, S; Ghosal, G. Fuel, v. 84, n. 5, p. 487-494 (2005)

Use of sugarcane bagasse to obtain structured aluminain the replica method for carbon and H_2 production

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Sugarcane bagasse (lignocellulosic material) is the most abundant solid residue from sugar and alcohol production, in which it is predominantly destined for burning to generate energy. In order to minimize this burning, many studies highlight an alternative use for this waste. Thus, in this work, the sugarcane bagasse was used in the replica method for alumina production with predetermined dimensions. Sugarcane bagasse samples were impregnated with an aqueous solution of $Al(NO_3)_3.9H_2O$, pressed at 13 kgf for production of cylindrical green bodies followed by drying at 75 °C and calcination at 1500 °C. The samples obtained after calcination were impregnated with different concentrations of Ni (5%, 10% and 20% w/w), resulting Ni anchored in structured alumina, Ni/Al_2O_3-S samples,followed by characterization by SEM, XRD, TPR, nitrogen fission at 77 K and TGA. In themicrographs, it was observed that Al_2O_3-S maintained the morphological characteristics of the test body and the alpha alumina phase presence (XRD). The Ni/Al_2O_3-S samples showeda reduction temperature in the range of 370 - 650 °C, without the presence of nickelaluminate (XRD). The samples were submitted to methane decomposition reaction at 500 °Cfor production of carbon and $H_{2(g)}CO_x$free. The tests were conducted in a continuous flowreactor with passage of the gas mixture of $N_2:CH_4$(6:1 v/v) in a flow of 35 mL. min^{-1}, for 30 min. The samples of $5Ni/Al_2O_3$-S, $10Ni/\ Al_2O_3$-S and $20Ni/Al_2O_3$-S showed conversion of methane into carbon yields 11, 45 and 49%, respectively.

Acknowledgements:

CNPq, CAPES and CHICO-Nano/UFPE.

$WO_3/BiVO_4$ side-by-side electro spun heterojunction for photoelectrochemical water splitting

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The generation of hydrogen fuel by a photoelectrochemical cell (PEC) can be considered a green hydrogen production route when compared to other processes [1]. In this work, we reported the preparation of photoanodes based on $WO_3/BiVO_4$side-by-side fibers on FTO. Firstly, 0.2 g of Polyvinylpyrrolidone (PVP, Mw 1300000) was dissolved in 0.6 mL of ethanol and 2.0 mL of DMF with vigorously stirring for 2h. Then, 1.0 g of $WCl_6$was added to the above solution and continued to be stirred for 1h to form a blue solution. [2]. The $BiVO_4$precursor spinning solution was prepared adding 1.21 g of $Bi(NO_3)_3$. $5H_2O$, 0.35 g of PVP, and 0.66 g of $VO(acac)_2$to 1.0 mL of acetic acid, 1.6 mL of ethanol, and 1.4 mL of DMF, which was then stirred for 2h to form a green solution [3]. The two solutions were transferred to syringes for side-by-side electrospinning, under 15 kV, total rate of 0,36 mL/h, 18 cm from the collector, with ambient temperature and relative humidity at 20 °C and 45 %, respectively. After, the samples were calcined at 500°C for 1h (2°C/min). Fiber morphology and structure were studied using X-ray diffraction and scanning electron microscope. The photocurrent activities were evaluated by voltammetry and chronoamperometry in Na_2SO_4(0.1 molL-1) under visible light and interfacial properties by electrochemical impedance spectroscopy. Results demonstrate the $WO_3/BiVO_4$nanofibers formation and photocurrent density can be up to 80 μAcm^{-2}at 1.2 V vs. Ag/AgCl. The high photoelectrocatalytic properties could be attributed to the successful establishment of $BiVO_4$and WO_3heterojunction with lower resistance to charge transfer and higher transmission efficiency compared with pure $WO_3$and $BiVO_4$fibers.

Acknowledgements:
To LADEMA, GMPC, LMEM, LARX and CNPq for funding (Proc. 142243/2019-1).

References:
[1] Dong, Z. Int. J. Hydrog. Energy., vol. 47. p. 728-734 (2022)
[2] Song, K et al. Chem Electro Chem, vol. 5, p. 322-327 (2018)
[3] Liu, J. Mater. Chem. Phys. vol. 247. n. 122858 (2020)

J-Materials for application in smart devices for energy storage and conversion

Crystal growth from the melt: some fundamentals of growth and applications for energy conversion

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Advanced functional materials are fundamental to high-efficiency energy conversion devices. In particular, compared to polycrystalline bulk materials, single crystals present much higher physical properties thus, being of a strong interest in the high technology industry. The crystal manufacturers demand, in turn, the production of crystals with highpurity, a high degree of perfection, large size, and industrial-scale mass production. Fromthe basic science point of view, single crystals are also excellent systems for investigating fundamental principles and for discovering novel properties. In this context, over the last100 years, oxide bulk single crystals produced by different methods have been massively used for many purposes. In this work, it will be reviewed some fundamental aspects of boththe heat transfer theory and mechanisms of growth of oxide bulk single crystals producedby vertical Bridgman-Stockbarger (VB-S) route. It will be shown that such route demandsoptimized growth conditions to produce high-quality crystals for achieving higher conversion efficiency materials. Such conditions include a complete previous analysis of thethermal properties of the precursor oxides, the characterization of the temperature gradientin the region of the liquid-solid interface, a suitable choice of the dimensions of the crucible,and the growth rates, among other issues. In particular, such approach will be presented and discussed, focusing on the growth of ferroelectric single crystals. The optimization ofthe structural and piezoelectric properties as well as the potential induction of novel properties for energy conversion allowed in multiferroic/ferroelectric crystals by co-doping strategy will be also discussed.

Acknowledgements: The author would like to thank the financial support from FAPESP (grants 2017/13769-1; 2019/14688-0) and CNPq (grant 409780/2021-9).

Room-temperature optical and magnetoelectric response in A and B site co-doped layer-structured Aurivillius ceramics

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Ferroelectric oxide materials have been subject of innumerous investigations, due to their superior physical features, that make them ideal for developing new multifunctional materials. Complex ferroelectric oxides offer exceptional options to tune physical properties, such as optical and multiferroic, with high temperature stability. Among them, due to their lead-free nature, bismuth titanate (Bi4Ti3O12)-based oxides, belonging to the Aurivillius family, have recently gained interest viewing to improve photoferroelectric and magnetoelectric properties. Bismuth titanate structures, composed of three alternating (Bi2Ti3O10)2- perovskites layers sandwiched between two of fluorite-like bismuth oxide (Bi2O2)2+ layers, can present high stability even after significant chemical doping. In this work the influence of iso- and heterovalent doping in the structural, dielectric, optical absorption, ferroelectric, magnetic and magnetoelectric properties of Aurivillius structures (bismuth titanate-BIT based ceramics of Bi3.25A0.75Ti3-x(Co, Fe)x/2O12, where A=La, Nd or Sm and x=0, 0.1, 0.2, 0.3, 0.4), have been investigated. Single phase bulk ceramics were synthed through the conventional oxide mixture process. It was observed that heterovalent (Co, Fe) co-doping promotes a decrease in the band gap (red shift), compared with the Bi3.25A0.75Ti3O12 matrix, still maintaining a ferroelectric spontaneous polarization, and induce room temperature multiferroic state. These results are discussed and correlated with changes in the electronic structure, octahedral distortions (tilting angles and bond lengths), and oxygen vacancies formation, Acknowledgments: The authors gratefully acknowledge the Brazilian funding agencies CNPq and FAPESP.

The role of line defects on the physical properties of BiFeO3 nanoparticles

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Bismuth ferrite ($BiFeO_3$ - BFO) is a perovskite structured (ABO_3) multiferroic magnetoelectric compound that, due to its simultaneous ferroelectric and antiferromagnetic ordering at room temperature, is one of the most promising candidates for applications in advanced electronics. However, the presence of a magnetic spin-cycloid with a period of 62 nm suppresses its long-range magnetic response. Several strategies can be applied to suppress or break the spin-cycloid to release the BFO magnetization, as doping/substitutionof iso or heterovalent ions in specific sites of the perovskite cell, or nanostructuration. Inthis context, high-energy cryo-milling was recently applied for synthesizing BFO nanopowders with controlled crystallite s and internal strain, resulting in an unusual non-linear and enhanced magnetic response that differs from that widely reported for bulk BFO. In this contribution, the role of line defects, as edge and screw dislocations, on the overall physical properties of multiferroic magnetoelectric nanoparticles is discussed taking BFO nanoparticles as a study model. It is pointed out that the control of the dislocation density in 2D or 3D nanostructures can be used as efficient tool to control physical properties and tailor specific technological applications.

Application of the GPA tool in Ferroelectric Prototypes Analysis

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The tool, Geometrical Polarization Approach" (GPA), has recently been developed and usedto analyze the polar nature of Dielectric Materials with Perovskita structure and quantifytheir main resulting properties [1]. The GPA tool aims to reveal intrinsically characteristicsof polar matters still in powder, allowing to know their properties before the costly processto synthesize dense ceramic bodies. In this approach, the intrinsic polarization, as a signature of the polar state, is obtained from the off centering position of nominal negativeand positive electric charges into a unit cell, which is achieved from the relative atomicpositions of cations and anions by diffraction experiments [1]. Important characteristicshave been revealed comparatively through its use, such as polar responses to externalstimuli. In this work the GPA tool was applied to materials under extreme external stimuli,such as high pressures. Different ionic modifications were also investigated within the unitcells and their respective responses to polarization [2]. We analyzed complex ceramic, withmodifications in the cationic atomic sites of perovskites. Finally, different theoretical arrangements for Rietveld refinement were proposed to investigate local polarization situations and compare them with the global one.

Acknowledgements:
Fundação Araucária Apoio ao Desenvolvimento Científico e Tecnológico do Paraná

References:[1] V.F. Freitas, J.A. Eiras, L.F. Cótica, and I.A. Santos, Ceramics International 47, 943-952 (2021). [2] V.F. Freitas, J.A. Eiras, L.F. Cótica, I.A. Santos, Ceramics International 48, 11805-11811 (2022).

Developing a Highly Sensitive Sensor for Low Intensity and Low Frequency Magnetic Fields using $CoFe_2O_4$ and PZT Fibers

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The magnetoelectric sensor is a class of magnetic sensors with high sensitivity that suitable for the measurement of low-frequency weak magnetic fields. This study presents the synthesis and characterization of a magnetoelectric composite material composed of $CoFe_2O_4$and PZT fibers, with the aim of developing a sensor for low intensity and low frequency magnetic fields [1]. The composite exhibits a strong magnetoelectric coupling, allowing for the conversion of a magnetic field signal into an electrical signal. The resulting sensor is highly sensitive and able to detect magnetic fields with low intensity and frequency, making it ideal for measuring biological magnetic signals such as those generated by heartbeats. We demonstrate the feasibility of using this magnetoelectric composite as a magnetic field sensor through experimental measurements and simulations. Our results indicate that this sensor has great potential for use in medical applications, including non-invasive monitoring of cardiac activity. Overall, the development of this magnetoelectric composite provides a promising avenue for the advancement of magnetic field sensing technology.

The authors would like to thank Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and Fundação Araucária de Apoio ao Desenvolvimento Científico e Tecnológico doEstado do Paraná Brazilian agencies, for providing financial support.

Reference: [1] J. Wang, D. Gray, D. Berry, J. Gao, M. Li, J. Li, D. Viehland, Advanced Materials, 23, 4111 (2011).

Energetic and electronic properties of metal-organic frameworks doped with transition metals: A hybrid functional study

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Metal-organic frameworks (MOFs) are crystalline porous solids with large surface areas formed by coordination bonding between organic ligands and metal centers (ions or clusters). Owing to their structural diversity and functional adjustability, these nanoporous materials have been extensively investigated for applications in energy storage, gas separation, and sensing. Tens of thousands of unique structures have since been synthesized. However, only a few works on electronic structure and transport properties have been reported. Here, we use DFT hybrid functional calculations to explore the incorporation of transition-metal impurities in the iconic metal-organic structure Zn4O(BDC)3 (BDC=benzene-1,4-dicarboxylate), known as MOF-5 or IRMOF-1.[3] We focus on the electronic structure, formation energy, and conductivity of MOF-5 doped with transition metals M occupying a Zn site (M=Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, and Al), exploring a possible doping mechanism. Our results show that MOF-5 is an insulator with a fundamental band gap of 4.8 eV, where the frontier orbitals, namely the valence band maximum (VBM) and the conduction band minimum (CBM) are associated with the ligands. The incorporation of metal impurities in MOF-5 introduces defect states in the band gap, changing its electronic properties while preserving the structure. Formation energy calculations indicate that Sc and Al are the most stable impurities exhibiting donor levels close to the conduction-band minimum, remarkably decreasing the band gap to around 0.2 eV. Whereas Cr, Fe, and Ni are stable in a neutral charge state, inducing midgap levels. Our results suggest that selected metal dopants can induce p-type conductivity. The control of the band gap and semiconducting properties observed will render MOF-5 a wide range of applications in electronic and optoelectronic, and sensing devices. This work was supported by the FONDECYT Project 1230138 and the Anillo Project ANID/ACT 210059.

Exploring the phase transitions of low-dimensionalmetal halide perovskites

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Low-dimensional metal halide perovskites have shown remarkable properties, making them promising materials for optoelectronic applications such as photovoltaics, light-emitting diodes, and lasers. In this contribution, we present recent studies on the phase transitions and properties of these materials using techniques such as single-crystal and synchrotron X-ray diffraction, as well as optical and vibrational spectroscopies, under varying temperature and pressure conditions. Distortions on the metal halide polyhedral network determine the excitonic states, favoring the production of self-trapped excitons (STEs). STEs play a crucial role in the phase transitions of these materials, especially in low-dimensional systems where confinement effects are more pronounced. The presence of STEs is correlated with the structural and optical properties, and their behavior depends on changes in temperature and pressure. Our results provide new insights into the relationship between the structureand electronic processes that lead to the unique optoelectronic properties exhibited by these materials. These findings can guide the optimization of perovskite-based optoelectronic devices by controlling the STE behavior.

1. Castro Ferreira, W.; Araújo, B. S.; Gómez, M. A. P.; Medeiros, F. E. O.; de Araujo Paschoal, C. W.; da Silva, C. B.; Freire, P. T. C.; Kaneko, U. F.; Ardito, F. M.; Souza-Neto, N. M.; Ayala, A. P. Pressure-Induced Structural and Optical Transitions in Luminescent Bulk Cs4 PbBr 6. J. Phys. Chem. C 2022, 126 (1), 541-550.

2. Rodríguez-Hernández, J. S.; P. Gómez, M. A.; Abreu, D. S.; Nonato, A.; da Silva, R. X.; García-Fernández, A.; Señarís-Rodríguez, M. A.; Sánchez-Andújar, M.; Ayala, A. P.; Paschoal, C. W. A. Uniaxial Negative Thermal Expansion in the [(CH 3) 2 NH 2]PbBr 3Hybrid Perovskite. J. Mater. Chem. C 2022, 10 (46), 17567-17576.

Growth and characterization of soft (K, Na)NbO$_3$-based single crystals produced by Bridgman-Stockbarger method

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Lead-based single crystals are widely recognized for their remarkable dielectric and piezoelectric properties. However, concerns about the impact of the extensive and continued use of Pb-based materials on the environment have led to the investigation of alternative piezoelectric compositions. Among these alternatives, lead-free single crystals based on (K, Na)NbO$_3$have shown great promise for substituting Pb-based materials [1]. In this study, soft (K, Na)NbO$_3$single crystals were grown using the Bridgman-Stockbarger method, and their physical properties were characterized. The XRD patterns of the crushed crystals showed a pure perovskite structure with an orthorhombic structure at room temperature, indicating a slight compositional shift compared to the precursor powder. The coefficients of thermal expansion of the single crystal were determined in different crystallographic directions using the dilatometric method for the first time, and the obtained values were compared to those of ceramics. The temperature dependence of the dielectric constant revealed two dielectric anomalies, corresponding to the ferroelectric-ferroelectric transition at 93 °C and the ferroelectric-paraelectric transition at 295 °C. The values of the dielectric constant at room temperature were found to be consistent with those reported in the literature for similar compositions.

Acknowledgments: The authors would like to thank the financial support from FAPESP (2019/26807-4 and 2017/13769-1) and CNPq (409780/2021-9).

References:
[1] WAQAR, Moaz et al. Evolution from Lead-Based to Lead-Free piezoelectrics: engineeringof lattices, domains, boundaries, and defects leading to giant response. Advanced Materials, v. 34, n. 25, p. 2106845, 2022.

Multidimensional Elemental and Molecular Analysis for Surface Interface studies

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Surface and Interface studies require the use of complementary analytical techniques as each instrumentation provides results based on the interaction of the investigated material with a probing medium [1]. Obtaining elemental and molecular information for different probing size and depth are especially crucial.HORIBA offers a Platform with multiple instruments able to tackle these complex analytical challenges. Glow Discharge relies on a plasma to sputter a representative area of a material and provides fast elemental depth profile with nanometer resolution [2]. Coupling GD and Raman microscopy allows to obtain molecular information at various depths with micrometer lateral resolution [3,4]. Applying the GD software ideal to follow transient signals to a simultaneous ICP instrument coupled with an electrochemical cell (AESEC technique) offers deep insight on dissolution mechanisms and metallic surfaces performances [5].We will illustrate the benefit of this Surface Platform for Elemental and Molecular Analysis with selected results on metallic parts for high temperature fuel cells, perovskite solar cells, hydratation of anodic films and DCL coatings on bipolar plates.

References:

1) Compendium of Surface Interface Analysis, Springer
2) Review: What Can Glow Discharge Optical Emission Spectroscopy (GD-OES) Technique Tell Us about Perovskite Solar Cells? Small Methods 2022, 2200633
3) Raman and glow discharge optical emission spectroscopy studies on structure and anion incorporation properties of a hydrated alumina film on aluminum. Applied Surface Science 592 (2022) 153321
4) Advances in RF Glow Discharge Optical Emission Spectrometry Characterization of Intrinsic and Boron-Doped Diamond Coatings. https://doi.org/10.1021/acsami.1c20785
5) Transient stainless-steel dissolution and its consequences on ex-situ bipolar plate testing procedure. International journal of hydrogen energy 45 (2020) 984-995

Multiferroic Coupling in $Pb(Zr_{0.2}Ti_{0.8})O_3/CoFe_2O_4$ Nanostructured Composites

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The magnetoelectric coupling in nanostructured systems has attracted interests mainly dueto possibility of integration between ferroelectricity and ferromagnetism through the handling of electric and magnetic order by cross coupling between electric and magneticfields. In this work, the fabrication process was investigated by the RF-Sputtering techniqueand the characterization of heterostructured multiferroic composite materials of Pb(Zr0.2Ti0.8)O3/CoFe2O4 were performed. The interface between phases has a significant role for the existence of coupling between the different phases, where variations of the ferroelectric and dielectric properties in the heterostructured system were correlated to strain and/or stress at the interface between the phases. It was also possible to determine the existence of magnetoelectric coupling, since an improvement of the ferroelectric properties was observed under the presence of a magnetic field of ~5 kOe. Finally, it was reported that the magnetodielectric coupling is enhanced in the dielectric dispersion region due to competition between the compressive stress in the PZT phase generated by CFO phase and the changes of electric polarization due to the ME coupling.AcknowledgementsThis work was supported by FAPESP (Grant numbers: 2020/06570-7 and 2017/13769-12) and CNPq (grant # 404538/2021-5).

Optimizing Parameters for $BiFeO_3$ Thin Film Formation via Dip Coating Deposition for Photovoltaic Applications

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The advancements in thin film materials techniques have led to significant progress in understanding material properties, resulting in devices with increased durability, reduced waste material, and smaller size while maintaining high efficiency, especially for photovoltaic applications [1-3]. In this work, the deposition parameters involved in the dip coating technique were analyzed in search of a better way to obtain $BiFeO_3$(BFO) thin films for photovoltaic applications. The results of the parameter analysis showed that a better deposition is mainly related to the substrate cleaning method, drying temperature, and solution concentration. The optimized deposition parameters were used for a single-layer photovoltaic device, which exhibited a photoconversion of 0.98 mV. These results indicate that dip coating with optimized parameters is a promising route for thin film formation of BFO for photovoltaic devices.

Acknowledgments

This work was supported by CNPq and Fundação Araucária de Apoio ao Desenvolvimento Científico e Tecnológico do Paraná (Pesquisa Básica e Aplicada).

[1]. Shah, A. et al. Photovoltaic technology: the case for thin-film solar cells. science. 1999 Jul 30;285(5428):692-8.

[2]. Green, M.A. Consolidation of thin-film photovoltaic technology: the coming decade of opportunity. Progress in Photovoltaics: Research and Applications. 383-92. 2006

[3]. Sundaram, S. Shanks, K. Upadhyaya, H. Thin film photovoltaics. InA Comprehensive Guide to Solar Energy Systems. Academic Press. 361-370. 2018

Optimizing Photoconversion Efficiency in $BiFeO_3$ Perovskite Thin Films through Temperature and Layer Control

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Over the past few years, perovskite materials have emerged as strong candidates for replacing traditional silicon solar cells due to their high efficiency and low cost [1]. The parameters for the fabrication of photovoltaic devices using these materials are directly related to their photoconversion efficiency [2,3]. In this work, thin films of multiferroic perovskite $BiFeO_3$were synthed by dip coating technique, and the photoconversion efficiency was found to be strongly related to the temperature of deposition and the number of layers. Specifically, an increase in the number of layers from 10 to 20 resulted in a significant increase in photoconversion efficiency, from 0.77 mV to 0.98 mV, respectively.

[1]. LI, H. et al. Photoferroelectric perovskite solar cells: Principles, advances and insights. Nano Today, Elsevier, v. 37, p. 101062, 2021
[2]. LOPEZ-VARO, P. et al. Physical aspects of ferroelectric semiconductors for photovoltaic solar energy conversion. Physics Reports, Elsevier, v. 653, p. 1-40, 2016.
[3]. YANG, M.-M. et al. Bulk photovoltaic effect in monodomain $bifeo_3$thin films. Applied Physics Letters, v. 110, n. 18, p. 183902, 2017.

Patrónite-based VS_4 Nanostructures: Fundamental Aspects and Applications in Lithium-Sulfur Batteries

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Patrónite is a mineral formed by vanadium tetrasulfides (VS_4) in a crystalline structure of quasi-one-dimensional nanowires bonded by van der Waals interactions. Recently, VS_4nanostructures were synthesized on substrates of carbon nanotubes doped with nitrogen or reduced graphene oxides, with potential applications in lithium-sulfur battery cathodes. $VS_4$is a highly polar chalcogenide with strong interaction with lithium polysulfides (LiPS). It acts on the anchoring LiPS fragments, reducing the shuttling effect and increasing the battery lifespan. In this work, we investigate the electronic, magnetic, mechanical and structural properties of nanostructures based on VS_4. Intrinsic magnetic orders were observed in the nanowire, with phase transitions with plastic deformations. We also investigated the formation of native point defects in the VS_4nanostructures and LiPS bindings in these point defects. We show that vanadium tetrasulfides have interesting characteristics for applications in electronic and electrochemical devices, such as sulfur composite components in the cathode of lithium-sulfur batteries.

Acknowledgements

We thank MackPesquisa (Grant 221018), CNPq (Grant 311324/2020-7) and FAPESP (Grant 2022/14549-3) for financial support. We also thank computational facilities provided by the High Performance Computing Center (NACAD) at COPPE/UFRJ.

PVDF-PLA-HA COMPOSITES OBTAINED BY 3D PRINTING FOR APPLICATIONS IN PIEZOELECTRIC BIOMATERIALS.

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Polymers such as poly(vinylidene fluoride) (PVDF) and poly(lactic acid) (PLA) have been widely studied as biomaterials due to their biocompatible properties in contact with living tissues. In particular, PVDF presents piezoelectricity when in the β phase[1], such an effect favors bone regeneration and is even associated with the natural repair process of this tissue in living organisms [2]. When mixed with hydroxyapatite (HA), PVDF and PLA can form biphasic and triphasic composites with some electrical and biological properties similar to the bone tissues being potential candidates to be used as 4th generation of biomaterials in orthopedics and orthodontics. In the present work, composites of PVDF, PLA and HA in different concentrations were produced by 3D printing.Scanning electron microscopy (SEM) images indicate the formation of immiscible polymeric blends for the PVDF and PLA composites, which is altered with the addition of HAp in the sample, changing the blend to miscible. Furthermore, the addition of HAp promotes the bioactivity of the materials, which can be evaluated by SEM and Energy Dispersion X- Ray Spectroscopy (EDS). In addition, the printed samples have low contact angle indicating a high hydrophilicity of the material. Therefore, the composite presents great potential to be used in the future in biomedical applications.

References:
[1] JACOB, J. et al. Piezoelectric smart biomaterials. Inflammation and Regeneration, v. 38, n.2, p. 1-11, 2018.
[2] RUAN, L. et al. Properties and applications of the β phase poly (vinylidene fluoride). Polymers, v. 10, n. 3, p. 228, 2018.(JACOB et al, 2018).

Size and Dislocation Density-Dependent Magnetoelectric Effects in Cryomilled BiFeO3 Nanoparticles for Advanced Electronics and energyharvesting

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Bismuth ferrite ($BiFeO_3$ - BFO) is a perovskite-structured multiferroic material that exhibits simultaneous ferroelectric and antiferromagnetic ordering at room temperature, making it a promising candidate for electronic applications. The magnetoelectric coupling in BFO arises from the complex interplay of spin, lattice, and orbital degrees of freedom. The magnetoelectric effect in BFO nanoparticles can be influenced by factors such as particle size, microstructure, and defects or impurities. Smaller particle sizes result in increased surface effects that impact the properties and coupling of magnetic and electric order parameters. Moreover, defects or strain can alter these interactions, leading to changes in magnetoelectric coupling strength. Cryomilling has been employed to synthesize BFO nanopowders with controlled crystallite sizes and dislocation density, resulting in an unusual non-linear and enhanced magnetic response. In this study, cryomilling was utilized to achieve low crystallite sizes and introduce high dislocation densities. Atomic Force Microscopy (AFM)-based techniques, such as Piezoresponse Force Microscopy (PFM) (with and without an external magnetic field) and Magnetic Force Microscopy (MFM), were applied to investigate the effects of size and dislocation density on ferroelectric, magnetic properties, and magnetoelectric coupling. This comprehensive investigation provides insights into the role of size and dislocation density in modulating the multiferroic properties of BFO nanoparticles and their potential applications in advanced electronic devices.

Spray pyrolysis as an alternative route to producing thermoelectric layers of Na_xCoO_2

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Alternative energy sources and energy storage are currently essential issues for human development. Access to new technologies heavily relies on their maintenance, availability, and production costs. Additionally, an effective energy storage system and the generation of renewable energy work together to provide long-term solutions for the goal of a sustainable energy plant. As a result, one way to produce clean energy is by thermoelectric conversion of waste heat. We present a systematic analysis of thermoelectric oxide coatings produced by spray pyrolysis. Although it produces rough layers compared to other processes, this technology is cost-effective and adaptable for large-scale production. It is possible to develop polycrystalline $NaCoO_2$thin films at lower and higher crystalline conditions by controlling synthesis parameters, such as the molarity M of the precursor solution. Additionally, the annealing in the oxygen atmosphere improves the crystallinity and reduces the influence of oxygen vacancies. According to X-ray diffraction and Raman spectroscopy, the growth of γ-$NaCoO_2$phase maintains its crystalline symmetry even at high Na contents. Due to optical resonance conditions, Raman modes linked to oxygen in-plane motion can be seen depending on the excitation wavelength. The Seebeck coefficient and electrical resistivity fall as Na concentration rises in lower crystalline samples. As a result, when x= 1.5, a power factor of up to ~3,3 $\mu W/K^2m$ is achieved. Higher crystalline films have Seebeck coefficients up to 500 µV/K but with poor electrical conductivity.

Acknowledgments: Fundación Carolina

Strategies for incorporation of polypyrrole as pseudocapacitive component in hybrid supercapacitors

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The requirement for energy storage devices with mutual high energy and power density reinforced the research in new supercapacitors that combine pseudocapacitive components and electrical double-layer capacitance (EDLC). Polypyrrole is a conducting polymer with promising applications for wearables and for the Internet of Things due to its superior electrical properties. In particular, for the development of electrodes, the low capacitive retention of polypyrrole requires the incorporation of carbonaceous materials as additives. Herein, it is reported the result of systematic studies [1-3] that considered the incorporationof activated carbon, graphene oxide, carbon dots and modified carbon templates by the adequate N-doping process. The effect of the surface modification of activated carbon with polypyrrole was observed from the increase in the energy density of the devices in the order of 175% while incorporating good capacitance retention of the carbon derivative (from values in the order of 60% for pure polypyrrole to near negligible degradation at 1,000 cycles at high current density in hybrid devices). The strategy of mutually doping the carbonaceous substrate before the polymerization shows to be a promising step to reaching outstanding performance in hybrid supercapacitors with the incorporation of nitrogen-based groups on activated carbon returning areal capacitance in the order of 421,4 mF cm-2 and superior capacitance retention (characteristic of carbon derivatives). Acknowledgement: Brazilian Funding Agencies FACEPE, FINEP, CAPES, and CNPq.

References:

[1] G.S. dos Reis, R.M.A. Pinheiro Lima, S.H. Larsson, C.M. Subramaniyam, V.M. Dinh, M. Thyrel, H.P. de Oliveira, J. Environ. Chem. Eng. 9 (2021) 106155.

[2] J.D. Orlando, R.M.A.P. Lima, L. Li, S.A. Sydlik, H.P. de Oliveira, ACS Appl. Electron. Mater. 4 (2022) 5040-5054.

[3] R.M.A.P. Lima, M.C.A. de Oliveira, H.P. de Oliveira, SN Appl. Sci. 1 (2019) 325.

3D PRINTING OF PVDF-PLURONIC BIOACTIVE COMPOSITES FOR USE IN BIOMATERIALS WITH PIEZOELECTRICITY

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Piezoelectric polymers, especially poly(vinylidene fluoride) (PVDF), have been receiving considerable interest in the use of prostheses and bone healing, due to their useful special properties, such as non-toxicity, interesting mechanical characteristics, high chemical resistance and remarkable piezoelectricity. In such context, obtaining composites of PVDF and hydroxyapatite (HA), can improve biocompatibility and help bone growth [1]. However, a drawback of using PVDF in medical applications is its hydrophobicity, which compromises cell adhesion and proliferation. In this case, an alternative is the use of pluronic, an amphiphilic copolymer used for the transport of substances and dispersion in physiological solutions, which can promote the formation of self-organized micellar arrangements [2]. Therefore, in the present work, PVDF-Pluronic-127 composites were processed by extrusion, obtaining filaments for 3D printing. The biocomposites were evaluated by scanning electron microscopy (SEM), contact angle analysis and FTIR. Through SEM, it was possible to observe the interaction between the polymers in the blends. The addition of Pluronic made it possible to reduce the contact angle, indicating a hydrophilicity profile of the printed biomaterials. FTIR analysis demonstrate the presence of the polar phase of the PVDF in the biocomposite. Therefore, the composite may have potential properties as innovative devices for biomedical applications.

Acknowledgements: CAPES.

References:

[1] BONADIO, T. G. M. et al. Polyvinylidene fluoride/hydroxyapatite/β-tricalcium phosphate multifunctional biocomposite: Potentialities for bone tissue engineering. **Current Applied Physics**, v. 17, n. 5, p. 767-773, 2017.

[2] MOKHTARI, F. et al. Recent advances of polymer-based piezoelectric composites for biomedical applications. Journal of the Mechanical Behavior of **Biomedical Materials**, v. 122, 104669, 2021.

Characterization of $BiFeO_3$-$PbTiO_3$ thin Films for photovoltaics applications.

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Solar energy is a rapidly advancing field of renewable energy, with ongoing efforts to enhance efficiency and reduce costs. Perovskite-based solar cells are emerging as a promising option, with recent developments indicating that their efficiency is now comparable to that of traditional silicon cells [1]. The multiferroic material $BiFeO_3$-$PbTiO_3$(BFPT) exhibits ferroelectric polarization, which is responsible for the photovoltaic effect in materials with no center of symmetry, making it a promising candidate for application in solar cells. In this study, BFPT thin films were synthesized using dip coating technique, resulting in a uniform deposition of the material [2-4]. The experimental parameters were optimized to develop a photovoltaic device that showed a response of 3.47 mV/cm².

[1] National Renewable Energy Laboratory. (n.d.). Best research-cell efficiencies. Retrieved May 1, 2023, from https://www.nrel.gov/pv/cell-efficiency.html
[2] V. F. Freitas, L. F. Cótica, I. A. Santos, D. Garcia, and J. A. Eiras. J. - Synthesis and multiferroism in mechanically processed BiFeO3-PbTiO3 ceramics. Journal of the European Ceramic Society. (2011).
[3] V. F. Freitas, I. A. Santos, E. R. Botero, B. M. Fraygola, D. Garcia, and J. A. Eiras - Piezoelectric Characterization of (0.6)$BiFeO_3$-(0.4)$PbTiO_3$Multiferroic Ceramics". Journal of the European Ceramic Society. 94, 754 (2010).
[4] Freitas, V.F., Astrath, E.A.C., Dias, L.C. et al. On the Characteristics of Perovskite Structured $BiFeO_3$-$PbTiO_3$Thin Films: Their Potential to Multifunctional Photovoltaic Applications. Braz J Phys 51, 1215-1223 (2021). https://doi.org/10.1007/s13538-021-00925-4

Characterization of the thermal properties of 0.70Pb (Mg1/3Nb2/3)O3-0.30PbTiO3 powder as precursorfor single crystal growth

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A large number of studies on (1-x)Pb($Mg_{1/3}Nb_{2/3}$)O_3-xPbTiO_3[(1-x)PMN-xPT] have shown that this system has excellent ferroelectric and piezoelectric properties, mainly for those compositions near the morphotropic phase boundary (MPB) with x=0.30[1]. Several techniques have been designed to grow PMN-PT single crystals in the vicinity of MPB to improve even more the physical properties. Among such techniques, the Bridgman-Stockbarger is certainly the most common one [2]. In order to have a clear understanding and control over the growth parameters to improve the quality of single crystals grown by this route, a prior knowledge of the thermal properties of the precursor oxides is crucial. In this work, the thermal properties of 0.70PMN-0.30PT powder, including thermal conductivity, thermal diffusivity, specific heat capacity, melting and solidification temperatures were investigated. From the thermal properties, the biot and peclet numbers were obtained and thus, compared with theoretical models proposed in the literature to appraise the behavior of the solid-liquid interface during the crystal growth. The structural and segregation coefficients of the as-grown PMN-PT single crystal were characterized and discussed based on the approach used in this work. Acknowledgement: The authors would like to thank the financial support from FAPESP (2022/05578-0 and 2017/13769-1) and CNPq (409780/2021-9; 307783/2020-0) FAPEMIG (APQ-01856-22).References:[1] Akhilesh Kumar Singh and Dhananjai Pandey (2003). Evidence for MB and MCphases in the morphotropic phase boundary region of (1-x)Pb($Mg_{1/3}Nb_{2/3}$)O_3-xPbTiO_3A Rietveld study. PHYSICAL REVIEW B 67, DOI: 10.1103/PhysRevB.67.064102[2] H. Luo, G. Xu, H. Xu, P. Wang and Z. Yin, Jpn. J. (2000). Compositional Homogeneity and Electrical Properties of Lead Magnesium Niobate Titanate Single Crystals Grown by a Modified Bridgman Technique Appl. Phys. 39,5581. DOI 10.1143/JJAP.39.5581

CsGeI3: a new ferroelectric Rashba semiconductor

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Ferroelectric Rashba semiconductors (FERSC) are a new class of multifunctional materials which integrates different subfields: (i) Ferroelectricity (granting the switchability of ferroelectric polarization by an electric field, traditionally exploited in non-volatile memory elements), (ii) Rashba effects (bringing in spin-degrees of freedom and usually exploited in logic semiconducting-based architectures), and (iii) Semiconductor Spintronics (granting the integration with existing semiconductor-based technology). One of the most interesting features of FERSC is the link between spin texture and ferroelectricity, allowing a non-volatile electric control of spin degrees of freedom. This peculiar spin-electric coupling can bring new assets into the field of electrically-controlled semiconductor spintronics. The present study is focused on the ferroelectric perovskite halide CsGeI3. Electronic structure and Rashba-like effects were systematically investigated by relativistic first-principles density functional calculations. The ferroelectric polarization, band structure, densities of states, and spin texture were calculated. The analysis of the results indicates that non-negligible spin-splitting, due to a purely bulk Rashba effect, occurs mostly near the conduction band minimum. The control of the spin-splitting amplitude by changing the ferroelectric polarization was theoretically demonstrated, as well as the complete reversal of the spin texture when the ferroelectric polarization is switched.

Acknowledgements:
L.G.D.S thanks Fundação Araucária for financial support under grant 051/2020.

Effect of electrodeposition temperature on the crystallinity of Bi2Se3 films grown on different substrates

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Bismuth selenide (Bi2Se3) is a binary compound with attractive properties for application in energy conversion systems, such as thermoelectric and photosensitive devices. In this study, Bi2Se3 thin films were grown by potentiostatic deposition on different substrates (Au, ITO, and Cr) at room temperature (RT) and heated electrolytes to evaluate the temperature effect on properties such as morphology and crystalline structure. As expected, the electrolyte temperature affects the deposition kinetics, accelerating the deposition rate. Differences in the crystalline phase and morphology were also observed with the increase in the temperature of the deposition process. The heating of the electrolyte enabled the formation of more compact films, with mirror-like aspects in the case of Au and Cr substrates. The formation of the pure orthorhombic phase was observed for Au substrates with a decrease in crystallinity with the increase of electrolyte temperature. The film growth on Cr substrates showed the coexistence of orthorhombic and rhombohedral phases, with a predominance of the orthorhombic one. The effect of temperature in this case improves the crystallinity of the deposited layers. The film growth on ITO substrates at RT conditions led to the formation of the rhombohedral phase, with the presence of characteristic peaks of the orthorhombic phase for the heated electrolytes. These observations can provide relevant insights for the adjustment of desirable physical properties of electrodeposited Bi2Se3 layers.

Effect of ionic substitution on the structure andproperties of the $CaTiO_3$ compound

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Calcium titanate ($CaTiO_3$), as a incipient ferroelectric, is a attractive material for photovoltaic applications, due to its optical and electrical properties, and for being heavy metals free (Lead Free). However, it does not have polar symmetry, a fact that prevents the bulk photovoltaic effect occurrence, characteristic of non-centrosymmetric materials. To overcome this problem, the iron ion was added to the compound through substitution, obtaining the compound $Ca[Ti, Fe]O_3$. In this work, we study the structure, electrical and magnetic properties of the material aiming to correlate structure-properties. Substitution with different amounts of iron showed significant differences in the structure of the material for photovoltaic applications.

Acknowledgments

This work was supported by CNPq and Fundação Araucária de Apoio ao Desenvolvimento Científico e Tecnológico do Paraná (Pesquisa Básica e Aplicada 09/2021).

References

[1] Bati, A.S.R., Zhong, Y.L., Burn, P.L. et al. Next-generation applications for integrated perovskite solar cells. Commun Mater 4, 2 (2023)

[2] Bandyopadhyay, A., et al. Effect of Iron substitution on structure and optical properties of nanocrystalline CaTiO3. Journal of Nano Research 3, 123-128 (2008)

Heat Treatment Effect on Physical Properties of Nb_2O_5 Nanoparticles

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Niobium pentoxide (Nb_2O_5) exhibits considerable potential for use in a wide range of technological applications. Its physical properties (e.g., structural, optical, electrical, etc.) are highly dependent on the synthesis method, chemical composition, and heat treatment conditions, resulting in the formation of different crystalline polymorphs [1, 2]. The present work investigates the influence of heat treatment on the physical properties of Nb_2O_5 nanoparticles. Heat treatments were performed at 300, 500, 800, and 1,000 °C for 1 hour, followed by cooling in ambient air outside of the furnace. Structural analysis was performed by X-ray diffraction (XRD). Morphological characteristics and particle size distribution were evaluated by transmission electron microscopy (TEM) and the optical properties themselves were determined via ultraviolet-visible spectroscopy (UV-Vis), from which band gap values were also determined using Tauc's plot. The results reveal that the crystalline structure of the non-heat-treated sample comprises an amorphous phase of Nb_2O_5 and monoclinic phase (H-Nb_2O_5). With an increase in temperature to 500 °C and 800 °C, a transformation occurs leading to a combination of H-Nb_2O_5 and orthorhombic phase (T-Nb_2O_5). Subsequently, at 1,000 °C, the structure is composed mainly of H-Nb_2O_5. The heat treatments induced changes in the morphological characteristics of the particles, and TEM images demonstrate that particle sintering occurs at 1,000 °C. The range of band gap was found to be around 3.62 eV for non-heat-treated samples and 3.33 eV for those heat treated at 1000°C. These values demonstrate the potential for application in electronic devices, such as photocatalysts.

References:

[1] Schäfer, H. et al., Angew. Chem. Inter. Vol. 5, 40 - 52, (1966).[2] Nico, C. et al., Prog. Mat. Sci., Vol. 80, 1 - 37, (2016).

Hyaluronic Acid Based Triboelectric Nanogenerator

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The recent development of the Internet of Things and wearables requires the integration of electronic devices and batteries/ supercapacitors with requisites of washability, flexibility and long-life cycle. Due to the restrictions of depletion/ periodic substitution of energy storage devices, the development of self-powered systems based on triboelectric nanogenerators applied in electronics introduces advances in wireless communication and biomedical fields[1]. As a consequence, the use of biocompatible and biodegradable materials has proven an outstanding increase due to the interest in wearables and implantable triboelectric nanogenerators. This study reports a novel and simple TENG based on hyaluronic acid and Ecoflex 00-30™ acting as tribopositive and tribonegative pairs respectively, resulting in a simple, low-cost, and easily integrated component for transparent and flexible devices, with output performance of voltage, current, and charge transfer of 398.48 V, 21.88 µA and 85.9 nC respectively. The device also proved to be capable of lighting up commercial LEDs and acting as a sensor of temperature. A calibration curve was evaluated at a fixed range of frequencies and the generated voltage proved an inverse relation with the temperature. An arduino-based system for processing the data was applied in the online identification of the temperature from the generated voltage in the TENG.

Acnowledgment: FACEPE, FINEP, CAPES and CNPq

References [1] I.C.M. Candido, G.D.S. Oliveira, G.G. Viana, F.A.G. Da Silva, M.M. Da Costa, H.P. De Oliveira, ACS Appl. Electron. Mater. 4 (2022) 334-344.

Influence of dispersant and freezing temperature on the pore structure of alumina monoliths manufacturedby the freeze-casting method

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In recent years, the freeze-casting process has stood out in the manufacturing of ceramic materials, due to the possibility of creating complex porous structures in a simple and efficient way [1-3]. In this work, α-Al2O3 monoliths were produced using tert-butyl alcohol solvent and parameters were evaluated such as dispersant influence, suspension agitation, and freezing temperature of the process. From the characterization techniques by microtomography, Archimedes'' principle, and electron scanning microscopy, it was possible to observe a total porosity of up to 60%, with the formation of prismatic porosity, with good connectivity between the pores and a decrease in alumina agglomerates when used citric acid dispersant and mechanical agitation of the suspension. The Fourier Transform Infrared Spectroscopy spectra results indicated maintenance of the α-Al2O3 structure and probable improvement in particle dispersion for the system with a freezing temperature of −170°C. In addition, compression tests demonstrated adequate strength in the range of 5 to 9 MPa. As shown, the characterization results corroborate the possible application of the ceramic material as a support for gas separation membranes [1,3].

Acknowledgments:
To the academic assistance of the advisor Prof. doctor Alysson M. A. Silva, for the support of the Microscopy Laboratory of the Institute of Geosciences at UnB and the Center for the Development of Nuclear Technology (UFMG) and for the financial support of Petronas, Finatec and DPG/UnB.

References:
[1]G. Shao, D. A. H. Hanaor, X. Shen and A. Gurlo, Advanced Materials vol. 32, 1907176 (2020). ;
[2]M. Q. Sun, P. Shen and Q. C. Jiang, Journal of Materials Science vol. 54, 5224-5235 (2019);
[3]D. F. Souza, E. H. M. Nunes, D. S. Pimenta, D. C. L. Vasconcelos, J. F. Nascimento, W. Grava, M. Houmard and W. L. Vasconcelos, Materials Characterization vol. 96, 183-195 (2014).

Integrating Machine Learning and Geometrical Polarization Approach for Predicting Electrical Polarization in Multiferroic Materials

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The pursuit of uncovering innovative materials with exceptional performance has become a pressing matter in the realm of the materials science. Traditional experimental and computational methods often involve substantial investments of both time and monetary resources, and are also constrained by a multitude of factors. This study explores the use of machine learning in materials science [1], specifically in obtaining electrical polarization information from BiFeO3 related materials based on crystallographic data. We demonstrate the effectiveness of machine learning in predicting the electrical polarization of these materials by training a neural network on a dataset of crystal structures and corresponding polarization values. The model achieved high accuracy in predicting the polarization of new materials, even those with complex crystal structures. Our findings highlight the potential of machine learning as a powerful tool in materials science research, enabling researchers to efficiently explore and predict properties of new materials. Intrinsic electrical polarizations were calculated using the Geometrical Polarization Approach" (GPA) [2].

The authors would like to thank Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and Fundação Araucária de Apoio ao Desenvolvimento Científico e Tecnológico doEstado do Paraná Brazilian agencies, for providing financial support.

References:[1] K.T. Butler, F. Oviedo, P. Canepa, Machine Learning in Materials Science, ACS, 2022. [2] V.F. Freitas, J.A. Eiras, L.F. Cótica, and I.A. Santos, Ceramics International 47, 943-952 (2021).

Nd^{3+}/Yb^{3+} co-doped sodium-calcium silicate glasses:down - and up-conversion processes

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Glasses containing sodium silicate co-doped with Nd^{3+} and Yb^{3+} ions were synthesized using the melt-quenching technique. The analysis was performed by varying the doping rate while keeping the concentration of the Nd^{3+} sensitized fixed at 0.2 mol% and the Yb^{3+} activator concentration at 1.0 mol%. The energy transfer mechanisms between Nd^{3+} and Yb^{3+} were analyzed by studying the energy levels involved and the relationship between the emission excitation power and its intensity. The results indicate that the presence of Yb^{2+} is likely in both Nd^{3+}-free and co-doped samples. The energy transfer was confirmed by the low conversion NIR emission spectra of the co-doped samples, which were excited at 808 nm. It appears that the observed mechanisms involve only entail the emission of a single NIR-absorbed photon, which occurs by direct energy transfer between ${}^4F_{3/2}Nd^{2+}$and ${}^2F_{5/2}Yb^{3+}$, with the assistance of the energy difference between these levels. The efficiency of energy transfer increases with the ytterbium content, reaching approximately 90% for the sample with 1 mol% Yb_2O_3, as determined by lifetime analysis. The excitation of trivalent neodymium (808 nm) and ytterbium (975 nm) ions leads to the observed up-conversion photoluminescence. The observed up-conversion emission bands observed are attributed to the 4f-4f transitions of Nd^{3+} and the forbidden 5d-4f transitions of Yb^{2+}. Under excitation at 975 nm, up-conversion of Nd^{3+} is observed, with an almost quadratic dependence of emission intensity on energy. This suggests that two laser photons are involved in the up-conversion process, indicating that the energy transfer occurs through the processes of assisted and cooperative energy transfer.

Work supported by: CAPES, CNPq, Fundação Araucária and FINEP.

On the structural, optical and polarization properties of the incipient ferroelectric $CaTiO_3$ compound

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Perovskites have been pointed out as promising candidates for several technologic areas, especially for photovoltaic applications. In this work, $CaTiO_3$compounds were synthed by using Pechini method. Their structural, optical and electric properties were investigated using X-ray diffraction technique, Rietveld analyses, FullProf and VESTA software. Tilted octahedral oxygen arrangements with similar center of symmetry for positive and negative electric charges are observed forming non polarized Perovskite structures. The origin of the structural observed distortions is directly linked to the strongest chemical bond between the ionic $O_1^{-2}$and Ti^{+4}sites. Direct (2.9 eV) and indirect (3.8 eV) optical band gaps are also observed. Two energy gaps indicate that this compound can absorb energy in two distinct bands, which is interesting for photovoltaic applications.

Acknowledgments

This work was supported by CNPq and Fundação Araucária de Apoio ao Desenvolvimento Científico e Tecnológico do Paraná (Pesquisa Básica e Aplicada 09/2021).

References

[1] M. D. Biegalski, et al., Impact of symmetry on the ferroelectric properties of CaTiO3 thin films. Appl. Phys. Lett. **106**(16), 162904 (2015).
[2] J. Rodriguez-Carvajal, Recent advances in magnetic structure determination by neutron powder diffraction. Phys, B: Condens. Matter, **192**(1-2), 55-69 (1993).
[3] V.F. Freitas, J.A. Eiras, L.F. Cótica, I.A. Santos, Geometrical polarization approach: A semi-empirical tool to estimate the intrinsic polarization of polar dielectrics, Ceram. Int. **47**(1), 943-952 (2021)

Optical properties of Tm^{3+} doped tellurite glasses

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Glasses doped with Tm3+ ions have been investigated for a practical near infrared phosphor [1]. These phosphors can be used in fiber gyroscopes [2], optical coherence tomography used for biological applications [3]. In the present investigation, we prepared tellurite glasses with 65TeO2-15Li2O-20ZnO and 60TeO2-15Li2O-25ZnO compositions and doped with different concentrations of Tm2O3 (0.5, 1.0 and 2.0 mol%). The purpose of the present work is to report the spectroscopic properties of Tm3+ ions in these tellurite glasses and to have a better understand of the effect of the glassy network on the behavior of Tm3+ ions in these hosts. The samples were synthetized by conventional melt-quenching method. The absorption spectra for Tm3+ were recorded in the UV-VIS and NIR regions. Emission spectra in the visible and near infrared regions were obtained using a continuous argon ion laser and luminescence decay were assessed using nanosecond pulsed laser, both measurements under 476 nm excitation. Tm3+ characteristic emission bands were observed at 650, 805, 1200, 1450 and 1630 nm, corresponding to 3F2,3→3H6, 3H4→ 3H6, 1G4→3H4,3H4 →3F4, 3F4→3H6, respectively. The assigned energy level data of Tm3+ in these glasseswere analyzed and the measured oscillator strengths of the absorption bands, the intensity parameters, Ωλ were calculated by using the Judd-Ofelt theory [4,5]. Using these data, radiative transition probabilities, radiative lifetimes, branching ratios and the integrated absorption cross-section for VIS and NIR transitions were calculated. From these analyses it was possible to indicating slight environmental differences at the Tm3+ ions for the different compositions of tellurite glasses.

[1] S. Nishimura, et al., J Mater Sci: Mater Electron. 28, 7157 (2017)[2] C.C. Cutler, et al., Opt. Lett. 5, 488 (1980)[3] A.F. Fercher, et al., Rep.Prog. Phys. 66, 239 (2003)[4] B.R. Judd, Phys. Rev. 127, 750 (1962); [5]G.S. Ofelt, J. Chem. Phys. 37, 511 (1962).

Optimization of the manufacturing process of BFPTthin films by immersion using rheological analysis

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Photovoltaic energy conversion has daily intensified the interest in new compounds that present photovoltaic responses combined with low production cost, as well as leveraged the creation of devices on nanometric scales for this purpose. In the quest to circumvent the high production cost and make solar cells more accessible, the use of thin films to compose photovoltaic cells has expanded. Among them, the compound (x)BiFeO3-(1-x)PbTiO3 (BFPT) has been gaining prominence for being a promising multifunctional material with regard to the production of devices for the collection and conversion of energy, as it manifests electrical, magnetic and piezoelectric properties arising from its composition. The BFPT resins manufactured for this work were synthed by chemical route (Pechini method) and deposited on platinum and ITO conductive substrates using the immersion technique. The characterization of the resins and films were performed by rheology and scanning electron microscopy (SEM), in order to quantify their behavior, correlating it with the water control distributed between the resins, to optimize the production process and final cost of the films. The results of the rheological analyzes made it possible to relate the physicochemical properties of the resin with the deposition speed and final thickness of the thin films, by providing information on viscosity, elasticity and behavior over time under the effects of temperature, pressure, shear rate and composition of the resin applied to the material, which allowed a controlled parameterization.

Acknowledgments: To those who contributed to the discussion of this work.

References:

[1] Hiratsuka, RS, Santilli , CV, Pulcinelli , SH (1995). The sol-gel process: a physical-chemical view. New Chemistry , 171-180.

[2] Livage , J., Henry, M., Sanchez, C. (1988). Sol-gel chemistry of transition metal oxides. Progress in solid state chemistry , 18 (4), 259-341.

Photophysical investigation of deep-red emitting PMMA films doped with Ir^{III} complexes: designing an active layer for luminescent solar concentrators

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The improvement of energy efficiency of buildings can be achieved by utilizing renewable energy sources, like sunlight[1]. Luminescent Solar Concentrator (LSC) has emerged as a solution for integrating photovoltaic (PV) devices into the structure of buildings. Therefore, the use of LSCs allows the use of solar energy through the coupled LSC-PV device and the harmonization with the local architecture. Luminescent materials can be used for LSCs[2], as they absorb the higher energy incident solar radiation and convert it into a more suitable lower energy radiation for the c-Si PV. Heteroleptic iridium(III) complexes are good candidates for LSCs for gathering the properties an LSC needs. Here, we report the synthesis and photophysical studies of three complexes, $[Ir(C\hat{}N)_2bqdc]$ ($C\hat{}N$=ppy, phq, and bzq), ppy=2-phenylpyridine (Ir-pb), phq=2-phenylquinoline (Ir qb), bzq=benzo[h]quinoline (Ir bb), and bqdc=potassium 2,2''-biquinoline-4,4''-dicarboxylate, that were incorporated into PMMA films varying the doping at 0.1, 0.25, 0.5, 0.75, and 1 wt% via the drop-casting technique on glass substrate. Through UV-Vis spectroscopy, intense absorptions between 300-600 nm were observed, originating from the ^{1,3}LC and ^{1,3}MLCT states. By Photoluminescence a wide emission band was obtained in the deep-red which is in resonance with the maximum optical response of the c-Si PV cell. The 1.0wt% films presented maximum emission at the deep-red, long lifetimes, and good quantum yields: PMMA:Ir(bb): (638 nm; 780 Φ 8.6 %), PMMA:Ir(qb): (650 nm; 806 Φ 1.4 %), and PMMA:Ir(pb): (660 nm; 950 Φ 10.6 %). Herein, we conclude that complex Ir-pb exhibited better properties for future LSC applications since it has the highest Φ, the longest lifetime, and the deepest red emission.

Thanks to CAPES, and FAPESP (19/26103-7).

[1] R. Rondão, et al. Nanoscale Adv. 9 (2023).
[2] K. Wu, H. Li, et al. Nat. Phot. 2, 101-110 (2018).

PROCESSING OF FLEXIBLE BATIO$_3$/PVA PIEZOELECTRIC MEMBRANES, FOR USE IN ENERGYCONVERTERS

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In recent years, piezoelectric materials have motivated interest in energy harvesting due to their ability to convert mechanical energy into electrical energy. In this work, the composite of barium titanate ($BaTiO_3$) and polyvinyl alcohol (PVA) was prepared by the solvent casting method, with a ratio of 1:1 in relation to the amount of solvent used. The diffractogram (XDR) shows that the peak around 20° of the sample refers to the PVA, and the peaks around 22°, 31°, 38°, 45°, 51°, 56°, 65° and 75 ° are compatible with the XRD of $BaTiO_3$, as a result, the nature of both materials prevailed in the produced membrane. The polymer-ceramic interactions were analyzed by infrared spectroscopy (FTIR) where the structural characteristics were observed through the spectrum and the polymer and ceramic bands were identified. The electrical characterization of the sample was performed by complex impedance spectroscopy, from which a superposition of conduction processes associated with the two materials can be observed. The response of the conversion of mechanical energy into electrical energy was made by an electromechanical device which presented a voltage of approximately 40 mV, which can increase or decrease depending directly on the pressure applied to the device. As a result, the piezoelectric response of the synthesized $BaTiO_3$/PVA composite is confirmed.

Acknowledgements:
LPMAT research group, INDT and FAPEAM.

References:
[1] Y. Zhao; Q. Liao; G. Zhang; Z. Zhang; Q. Liang; X. Liao; Y. Zhang. Elsevier, page 1-9, 8 dez. 2014.
[2] C. Cerconi; P.P. González-Borrero. ISSN 1517-7076 artigo 11521, page 1510-1524, 2013.
[3] R. Shi, J. Bi, Z. Zhang, A. Zhu, D. Chen, X. Zhou. Carbohydrate Polymers, 74 V. page 763-770 (2008).

Production of α-Al2O3 macroporous ceramic materials in Naphthalene-Camphor solvent by the Freeze-Casting technique

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Freeze-Casting is a simple, versatile and very promising process for the manufacture of macroporous ceramic supports for application in gas separation. The technique is based on four steps: slurry preparatio controlled solidification of the slurry, sublimation of the solvent and sintering of the green body. [1,2] The porosity of the sintered material mimics the arrangement of the original solvent crystals, therefore, the shape of the pores is dependent on the nature of the solvent used. [3] This work proposes the study of densification (varying the sintering time), porosity, and mechanical resistance of α-alumina monoliths (α-Al2O3) in three different compositions of naphthalene-camphor solvent - hypoeutectic, eutectic and hypereutectic. The tests showed that, in general, the monoliths presented an average of total porosity of 63.6%, however without clear trends with the change of solvent composition or alteration of the sintering time. Finally, compression tests were performed on the samples to assess the mechanical strength of the material. The normal compressive stresses for each system ranged between 1.53 MPa and 23.56 MPa, and it is possible to observe trends according to the variation in sintering time and material composition.

Acknowledgments: I would like to thank the DPG/UnB, the Foundation for Scientific and Technological Enterprises (Finatec) and Petronas company for allocating resources and encouraging a research grant for this project. To the Laboratory of Geodynamic and Environmental Studies (UnB), the Nuclear Technology Development Center (CDTN-UFMG), and the Microscopy Center of UFMG for the analyzes and results carried out for the production of this work.

References:

[1] Deville, S. Advanced Engineering Materials, 10, 155-169 (2008);

[2] Nelson, I.; Naleway, S. E. Journal of Materials Research and Technology, 8, 2372-2385 (2019);

[3] Lacerda, L. D.; Souza, D. F.; Nunes, E. H. M.; Houmard, M. Ceramics International, 44, 16010-16016 (2018).

Production of cubic tungsten carbide films by radiofrequency right angle magnetron sputtering atroom temperature

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Tungsten carbide is widely used in biomedicine, optics, mechanical, aerospace engineering andrenewable energies [1], due to the different properties such as thermal stability at hightemperatures, good electrical conductivity, high hardness, resistance to oxidation, among others. Of all WC crystalline structures, the cubic system (β-WC) presents greater instability, making itmore difficult to synthesize in thermodynamic equilibrium processes, showing limitedapplications due to this difficulty. In this work, we synthesized thin films of tungsten carbide byright angle magnetron sputtering (RAMS) for the first time. The RAMS system has been widelystudied for the production of thin films of complex crystal structures as hydroxyapatite andsubstituted hydroxyapatite [2]. Our results show a production of crystalline cubic WC prepared atroom temperature. These results are confirmed by grazing incidence X-ray diffraction (GIXRD)and Rietveld refinement, high-resolution transmission electron microscopy (HRTEM), X-rayphotoelectron spectroscopy (XPS), RAMAN spectroscopy and density function theory (DFT).

Acknowledgements:The authors would like to thank the Brazilian Center for Research in Physics and Coordination forthe Improvement of Higher Education Personnel (CAPES) for the doctoral scholarship.

References:[1] U. Sohail, E. Pervaiz, M. Ali, R. Khoa, A. Shakoor, U. Abdullah, FlatChem 35, 100404 (2022).

[2] E.O. López, F.O. Borges, A.M. Rossi, R.M.O. Galvão, A. Mello, Vacuum 146, 233-245 (2017).

PRODUCTION OF RENEWABLE CLEAN ENERGY FROM TRIBOELECTRIC NANOGENERATORS PRODUCED WITH SHEETS OF CARBON NANOTUBES FUNCTIONALIZED WITH SILVER NANOWIRES

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A triboelectric nanogenerator (TENG) is a system that can convert mechanical energy into electrical energy at a small but steady pace. Self-powered and self-sufficient systems can largely benefit from TENGs, once they can be incorporated onto clothes, inside shoe soles and on car tires. The aim of this work is to prepare a single-electrode TENG capable of generating enough power to charge a capacitor or to power generic integrated circuit components and implantable devices. In the present work, Polyamide 11 and Polydimethylsiloxane (PDMS) were used as the triboelectric pair. A PDMS layer covered an electrode and the relative contact between the polyamide and the PDMS was responsible for generating the charges by contact electrification. The electrodes used were cotton fabrics functionalized with multi-walled carbon nanotubes (MWCNT), pure Buckypapers (BPs) and Buckypapers functionalized with silver nanowires (AgNWs) at different loadings (33.3 and 40 wt. %). Silver nanowires were used to further improve the BP's conductivity.The BPs were prepared by vacuum filtration of a MWCNT suspension in water and Triton X-100 andAgNWs with high aspect ratio were synthed in the laboratory through a polyol process. Thesamples were characterized morphologically by scanning electron microscopy (SEM). Conductivity tests were carried out on the samples of cotton, pure BP and BP functionalizedwith AgNW with and without the PDMS layer. The results showed that the electrical conductivity improved by increasing the AgNW content, as expected. The samples of BP +44wt.% AgNWs achieved the highest electrical conductivity and charge generated at thesurface of 1,4×103 S/cm and 4,33 pC, at room temperature. A higher content of AgNWs andother metallic nanostructures can increase even more these properties, making these materials very promising for engineering applications of low power demand devices.

Properties of Fe and Co doped KNN magnetoelectric ceramics

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Multiferroics materials in which the ferroelectric and magnetic properties coexist have been extensively studied in the last two decades for their versatility and possibility of application in an extensive range of electronic devices since their coupled order parameters.
Although mainly the magnetoelectric materials are based on systems with Pb2+ ions in their compositions, such as PZT, becoming a hazardous material since the beginning of its production. Alternatives using other intrinsic ferroelectric systems are the light for environmentally friendly material with similar properties to the regular magnetoelectric materials. In this case, using sodium and potassium niobate, $K_{1-x}Na_xNbO_3$perovskite ceramics becomes a promising solution strategy. In this work were synthed monophase magnetoelectric materials based on doping of KNN ceramics with Fe and Co oxides. The ceramics were densified using three different routes, and the properties were analyzed as a function of the doping and the densification route of the ceramic bodies. The results have shown significant changes in the densification properties in the doped system, mainly due to the site replacement and vacancy formations. All the ceramic bodies present an orthorhombic structure with no traces of secondary phases. The electrical properties show no significant changes in the fundamental properties of KNN, keeping the system with good ferroelectric properties. This work synthes all the properties of this system, looking forward to a new functional and friendly material for electronic applications.

Spectroelectrochemical studies of s-tetrazinederivatives and potential applications.

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S-tetrazine is a strong electron withdrawing heterocycle containing four nitrogen atoms in a six-membered ring. Many tetrazine derivatives exhibit emissive properties, moreover, their reversible electrochemical reduction often leads to fluorescence quenching. The intense color of s-tetrazine is due to the allowed n-π* transition in the visible range, which is a rare phenomenon. These features, combined with a low molecular weight, are the source of a growing number of scientific publications and potential applications of compounds containing 1,2,4,5-tetrazine[1]. The presented work includes electrochemical and spectroelectrochemical studies of D-A (donor-acceptor) and D-A-D (donor-acceptor-donor) s-terazine derivatives with carbazole electron donating units.

The tested monomers show weak fluorescence. A significant enhancement of the emissive properties was recorded for the soluble products of the electrode reactions. The introduction of a carbazole unit into the molecular structure enables the deposition of a layer by electrochemical oxidation. The obtained stable polymer films based on symmetrical s-tetrazine derivatives undergo electrochemical doping leading to reversible changes in the absorption spectrum and changes in the color of the layer. The results of the measurements indicate the possibility of their use in the production of electrochromic windows.

Acknowledgements: Financing by the EU Horizon 2020 grant no. 778158

References: [1] F. Miomandre, P. Audebert, Journal of Photochemistry and Photobiology C: Photochemistry Reviews vol. 44, (2020)

Structural and vibrational analyses in $PbTiO_3$ferroelectric thin films

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The use of ferroelectric materials in advanced electronic devices has increased the interest of such systems in high-performance practical applications [1, 2]. Indeed, their current integration in cell phones, ultrasound and medical instruments, sensors, electro-optical devices, as well as energy storage systems, has been possible because of their excellent physical properties with enhanced response in a very wide temperature and frequency interval [2]. In particular, the lead titanate ($PbTiO_3$, PT) ferroelectric system, which has a perovskite structure with tetragonal symmetry (P4*mm* space-group) at room temperature, stands out because of its very high spontaneous polarization, improved piezoelectric coefficient and relatively high Curie temperature [2]. However, because of the lead volatility during synthesis, it is difficult to obtain pure-phase PT system, avoiding the formation of secondary phases [3]. Therefore, for obtaining high quality samples, the synthesis process has to be carefully controlled. In this work, the physical properties have been investigated in PT thin films, obtained from the polymeric precursor’s method. The influence of the synthesis conditions on the structural and vibrational characteristics have been taken into account.

Acknowledgments: The authors thank to CNPq (309494/2022-2), FAPEMIG (PPM-00661-16 and APQ-02875-18) and CAPES (Finance Code 001) from Brazil, and NSF and ONR from USA, for the financial support.

References:

[1] K. Tadeuchi, Jpn. J. Appl. Phys. 28 105 (1989)
[2] Y. Wang, W. Chen, B. Wang and Y. Zheng, Materials 7, 6377 (2014)
[3] Y. Xu, Ferroelectric Materials and their Applications, Elsevier Sci. Publisher, The Nethelands (1991)

Study of electron transport properties in thin films of Nb_2O_5

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Niobium pentoxide (Nb_2O_5) has recently received increasing attention as an electron transport layer (ETL) in flat perovskite solar cells (PSCs) and is considered a possible alternative to replace titanium dioxide (TiO_2). In a typical PSC structure, it is composed of FTO/ETL/perovskite/HTL/metal electrode. The ETL is responsible for transporting the electrons generated by sunlight in the absorber layer to the external circuit of the cell [1]. Nb_2O_5 is a promising candidate as ETL in PSCs. This is partly due to the advances already achieved in energy conversion efficiency (PCE) using Nb_2O_5 as ETL, as demonstrated in [1], with PCE of 18.59%, in [2], with 17.2%, and in [3], with 20.22%. However, it is important to highlight that the synthesis and deposition method of Nb_2O_5 thin film plays a significant role in determining the PCE of PSCs [1]. Therefore, with the aim of obtaining an efficient material, it is necessary to investigate the electron transport properties in Nb_2O_5 thin films. In this sense, this work aims to investigate such properties in thin films manufactured through the Sol-Gel synthesis method, using dip-coating deposition techniques. It is expected to obtain an ideal material by optimizing the optical properties of the films to improve the efficiency of their electronic mobility.

Acknowledgment

Capes, CNPq, FAPERGS

REFERENCES

[1] FENG, J. et al. E-beam evaporated Nb_2O_5 as an effective electron transport layer for large flexible perovskite solar cells. Nano Energy, v. 36, p. 1-8, 2017.

[2] LING, X. et al. Room-temperature processed Nb_2O_5 as the electron-transporting layer for efficient planar perovskite solar cells. ACS Applied Materials Interfaces, v. 9, n. 27, p. 23181-23188, 2017.

[3] WANG, Z. et al. Solution processed Nb_2O_5electrodes for high efficient ultraviolet light stable planar perovskite solar cells. ACS Sustainable Chemistry Engineering, v. 7, p. 187-194, 2019

Study of the influence of freezing direction on pore morphology and mechanical strength in samples manufactured via freeze-casting

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Ceramic materials have advantageous properties for the production of supports used for gas separation. The freeze-casting method has great potential as a scalable manufacturing process for the production of porous ceramics, providing a material of controlled porosity[1-3]. The objective of this work was the production of ceramic monoliths, using camphene as solvent, and the evaluation of the influence of the freezing direction using different molds (copper, technyl, copper-technyl) on the pore morphology. The total porosity of the monoliths varied between 50-70% and this value was confirmed by the μ-CT analysis, it was evaluated that the synthesized monoliths obtained high mechanical resistance where the sample produced in the technil mold obtained the highest values. The SEM images showed that the samples produced in the mixed mold had a change in the perpendicular alignment of the pores in the transition region from copper to technyl [2-3]. In the copper mold, in turn, more aligned structures were observed forming continuous channels, and the technil dendrites did not present continuous alignment. Therefore, the methodology used for the technil mold presented a material with good porosity and ideal alignment of thesepores for application in gas separation.

Acknowledgments: Thanks to prof. Alysson Silva for guidance in carrying out this work. To the Laboratório de Estudos Geodinâmicos e Ambientais at UnB, the Centro de Desenvolvimento da Tecnologia Nuclear and the Centro de Microscopia at UFMG. And to the financial assistance provided by Petronas, Finatec and the DPG/UnB

References: [1] Shao, G., Hanaor, D. A., Shen, X., Gurlo, A. Advanced Materials vol. 32(17), 1907176 (2020). [2] Silva, A. M., Nunes, E. H., Souza, D. F., Martens, D. L., da Costa, J. C. D., Houmard, M., Vasconcelos, W. L. Ceramics International vol. 41(10), 14049-14056 (2015). [3] Arslanoglu, M., Ozdoganlar, O. B., Panat, R. Journal of the American Ceramic Society, vol. 105(8), 5114-5130 (2022).

Supercapacitor auto analyzer: An automated data analysis software for supercapacitor characterization

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High-performance supercapacitors (SCs) with high power, energy, and long-life cycles, are essential for innovating wearable and portable electronics as extensively reported in the literature [1]. The Web of Science shows more than 23,000 papers published since 2018 with the topic 'Supercapacitor', a number that keeps growing, from 732 papers per year in 2012 to an average of 4651 papers per year since 2018. However, the establishment of performance metrics on the reported values should be achieved to reduce inconsistencies in their real performances [2]. For that reason, we developed the Supercapacitors Auto Analyser (SCAA) program for SCs data analysis to support the community to establish performance metrics for SCs devices, with standard terms, equations, and units. The SCAA generates important graphs with the SCs key properties and electrochemical performance such as capacitance, energy, power, coulombic efficiency (CE), equivalent series resistance (ESR), drop potential (Vdrop), cycling stability, and time constant. These results are calculated from common data generated by potentiostat on SCs characterization such as cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). With appropriate inputs, the SCAA provides as many as 50 graphs with accurate normalizations by mass, area, or volume in a few minutes. The present work can speed up research on supercapacitors through automation of data analysis, packed in a software shared with the community and in the website www.scaasoftware.com.

Acknowledgments:
FAPESP (13/07296-2, 17/20809-0; 20/04721-8, 20/12356-8, 21/03379-7)

References:

[1] Poonam et al., Journal of Energy Storage, 21, 801 (2019)
[2] C.Arbizzani et al., Journal of Power Sources, 450, 227636 (2020)

SYNTHESIS AND CHARACTERIZATION OF POLYMERS BASED ON BENZOTHIOPHENE AND THIOPHENE WITH POTENTIAL APPLICATION IN PHOTOVOLTAIC DEVICES

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The growing demand for electric power has challenged present technologies and motivated researchers to find innovative and affordable solutions to solve this problem [1]. Polymeric solar cells (PSCs) have attracted academic and industrial interest due to their multiple advantages, such as flexibility and semi-transparency. Additionally, they are lightweight and can be produced on large scales at low cost by the industrial roll-to-roll method [2, 3]. In recent years, records of photovoltaic energy conversion efficiencies have been recorded. In order to produce more efficient conjugated polymers, this work aims to explore the synthesis and characterization of polymers with complementary absorption potential for application in a ternary device. The synthesized polymers are relatively simple and obtained from aromatic units of high electronic density: benzodithiophene (BDT) and thiophene (TH), using the Stille coupling reaction. A systematic study was carried out to verify the impact of the different side chains on the chemical and photovoltaic characteristics of these materials. The polymers obtained were characterized by 1H-nuclear magnetic resonance spectroscopy, thermogravimetric analysis, gel permeation chromatography, and UV-Vis spectroscopy. In addition, devices were produced using the synthesized polymers as a donor and PC71BM as an acceptor. For the production of this active layer, the influence of different rotations on the photovoltaic parameters of the devices was evaluated.

Acknowledgements:
To CNPq and FAPERJ for financial support.

References:

[1] P. K. Tawalare, Advances, 11, 100701 (2021).
[2] X. Guo, Q. Fan, J. Wu, G. Li, Z. Peng, W. Su, J. Lin, L. Hou, Y. Qin, H. Ade, Angewandte Chemie International Edition, 60, 2322-2329 (2020).
[3] P. M. Kuznetsov, I. E. Kuznetsov, I. V. Klimovich, P. A. Troshin, A. V. Akkuratov, Mendeleev Communications, 31, 30-32 (2021).

Synthesis and characterization of reduced nano-graphene oxide from oxidation of graphite

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Nano-graphene oxide (nGO) is an advantageous and up-and-coming nanomaterial based on its utilization in biomedical, chemical, electrical, optical, energy, and other areas [1]. In addition, its high electrical conductivity is one of the essential parameters for using this nanomaterial in renewable energy and electrical applications. Its electric conductivity is strong-willed by the suppression of dielectric areas of sp^3 carbon atoms linked to oxygen groups separating the planes associated with graphene [2]. Reduced nano-graphene oxide (nGOr) is obtained from the chemical modification of graphene [3]. Considering these essential characteristics of nGOr and Global warming in recent years, this research modified Hummer' s method to obtain nGOr. These nGOr were fully characterized by FTIR, UV Visible, XRD and SEM. FTIR showed chemical groups related to the graphite oxidation process at 3270, 2128, 1705, 1612 and 1032 cm^{-1}. In addition, UV-Vis presented bands at 233 and 300 nm associated to π-π* (graphitized core sp^3hybridization, LUMO→HOMO transition). XRD showed peaks at 25.05° related to graphite oxidation, and at 16.37° and 7.375°, indicating a decrease in layers of 0.541 nm and interplanar distance of 0.7 nm. SEM analysis showed several layers of leaf, resembling a crumpled sheet of paper with wrinkles and folds, due to interactions between oxygenated functional groups. These results demonstrated that the synthesized material presented an excellent quality, which will guide the realization of new syntheses to improve the results and demonstrate several applications for this nanomaterial.

Acknowledgment:
The authors thank FAPEMIG (APQ-02565-21), CAPES, CNPq and UFVJM for financial support to the project.

References:

[1] Dideikin AT, Vul' AY, Frontiers in Phys, v. 6, p.1-13 (2019).
[2] Neklyudov VV, Khafizov NR, Sedov IA, Dimiev AM, Phys Chem Chem Phys, v. 19, p.17000-8 (2017).
[3] D. Dorfey, S. R. Mortari and T. M. Volkmer, Disciplinarum Scientia 18, 421 (2017).

Synthetic Pathways Naphthalene Diimide (NDI)-bearing Polysilsesquioxane Gels and Their Impact on Photochromism

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This presentation will focus on organic/inorganic hybrid gels obtained by polycondensation of bis-alkoxysilanes incorporating naphthalene diimide bridges (**NDI**). The resulting **NDI**-based Polysilsesquioxane gels NDI-$[SiO_{1.5}]_2$ were found to exhibit interesting characteristics for many applications, in particular photochromic properties due to the photoproduction of **NDI** anion radicals. To understand the mechanism of photoreduction and its relation to the structure of the 3D network, we explored two different and orthogonal synthetic routes to the targeted polysilsesquioxane gels by studying their influence on the organization of **NDI** bricks within the network.[1,2] A one-step strategy has been developed to simultaneously generate "in situ" the **NDI** bridges and the polysilsesquioxane network (gel **1p**). The two-step strategy conversely required the prior synthesis of a siloxane-substituted **NDI** precursor which was eventually polymerized to form the targeted polysilsesquioxane-based gel material (gel **2p**). Both gels were thoroughly characterized by rheology, XRD/SAXS, spectroelectrochemistry, IR-ATR, Si NMR and impedance measurements. All these measurements suggest that **1p** and **2p** exhibit similar structural, optical and electrical properties.

Acknowledgements:

This work was supported by the São Paulo Research Foundation (FAPESP Grant n° 2019/27157-3 and n° 2021/06753-7). TBF also thanks the Ecole Normale Supérieure de Lyon (France) and USP.

References:

[1] PANJA, Santanu; ADAMS, Dave J. Chemical Society Reviews, v. 50(8), p. 5165-5200, 2021.

[2] MORAES, Thaisa BF et al. Journal of Luminescence, v. 204, p. 685-691, 2018.

The influence of sintering condition on the performance of superionic conductor ceramics

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The need for environmentally friendly devices has driven society to adopt renewable and non-polluting technologies, such as electric vehicles and small appliances powered by efficient and environmentally safe energy storage systems. However, this need increases consumption of raw materials to maintain and develop such technologies. Solid-state batteries and ion-selective membranes emerge as solutions to power devices and extract raw materials, respectively. In this context, the use of ceramic compounds as electrolytes is promising due to their high thermal and electrochemical stability, making them safer than traditional liquid electrolytes [1]. In addition, with its suitable electrochemical properties, ion selector membranes based on ceramic materials are relevant for seawater desalination and for the reuse/extraction of ionic and rare-earths species from batteries and other sources [2]. Those applications require high ionic conductivity, which can be achieved with superionic conductors. In this sense, this research aims to optimize the performance of superionic ceramic electrolytes by using laser-sintering technique. For this propose, $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$and $Na_{3.3}Zr_2Si_2PO_{12}$ceramics, with NASICON structure [3], have been prepared by solid-state reaction and sintered by conventional electric furnace for comparison purposes. Preliminary results show ionic conductivity in the range of 0.1-0.9 mS cm-1 at room temperature, which is expected to be improved when sintering using CO_2laser is carried out, due to the microstructure produced by high heating rates.

Acknowledgements:

To CNPq for the financial support

References:

1. YU, X et al., Engineering, vol. 21, pages 9-14(2022).
2. HOFS, B., et al., Separation and Purification Technology, vol 79, pages 365-374(2011)
3. ZHANGm, L., et al., Interdisciplinary Materials, col. 2, pages 91-110(2023)

Theoretical insights on the diffuse phase transition in ferroelectric materials

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Ferroelectric materials have been widely investigated over the last decades because of their excellent physical properties, which make them promissory systems for practical applications [1]. In particular, relaxor ferroelectrics with diffuse phase transition have played an important role in electronic devices such as capacitors, sensor, transducers andothers. However, the real mechanism on the diffuse phase transition (DPT) characteristicsremains unclear up today. Therefore, there has been an increasing interest in the scientificcommunity for a careful study of the physical properties of such system in order to betterelucidate the observed intriguing and unsolved phenomena. In fact, although several phenomenological methods have been proposed in the literature to account for the nature of the enhanced diffusivity of the paraelectric-ferroelectric phase transition in such materials [2, 3], the real mechanism still remains a matter of strong controversy. In this work, a macroscopic statistical model, which describe the dielectric response in relaxor systems with diffuse phase transition (DPT) [3], has been used to investigate the dielectric response in the (Pb,La)(Zr,Ti)O_3(PLZT) system, for a composition with relaxor behavior and DPT characteristics. The fitting of the experimental data has been performed via computer simulation using the Monte Carlo (MC) method, and implemented in python programming language.

Acknowledgments: The authors thank to CNPq (309494/2022-2), FAPEMIG (PPM-00661-16 and APQ-02875-18) and CAPES (Finance Code 001) from Brazil, and NSF and ONR from USA, for the financial support.

References:

[1] Y. Xu, Ferroelectric Materials and their Applications, Elsevier Sci. Publisher, The Netherlands (1991)

[2] V. V. Kirillov and V. A. Isupov. Ferroelectrics 5, 3 (1973)

[3] J. Liu et al., Phys. Rev. B 96, 054115 (2017).

White light source and optical thermometry based on zinc-tellurite glass tri-doped with $Tm^{3+}/Er^{3+}/Sm^{3+}$

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The study of the variation of luminescent properties with temperature is one promising way to measure temperature in many scientific and technological areas because it allows the determination of the same locally and contactless. Most optical thermometers utilize a ratiometric approach between thermally coupled levels [1-3]. However, such sensors have an inherent limitation of relative thermal sensitivity linked to the maximal energy gap between these levels, which can make them useless for some important applications. This work reports the potential application of $Tm^{3+}/Er^{3+}/Sm^{3+}$tri-doped TeO_2-Li_2O-ZnO (TLZ) glasses as temperature sensors using emitted color and luminescence intensity ratio (LIR) that do not have this limitation. The samples were successfully synthesized by melting quenching technique in an air atmosphere. We have used a fixed concertation of Tm^{3+}(0.2 mol%) and Er^{3+}(0.5 mol%) ions, and three Sm^{3+}concentrations: 0.2, 0.5 and 1.0 mol%. Particularly, we have analyzed the fluorescence intensity ratio (FIR) corresponding to the emissions of these ions, as a function of the temperature. As a result, the highest value obtained to the relative thermal sensitivity was 2.47 $\%K^{-1}$for the FIR related to the emissions at 530 nm and 458 nm, and for the sample doped with 0.5 mol% of Sm3+. In addition, white-light emission was achieved by adjusting the doping concentration of Sm^{3+}ion. Moreover, we obtained CIE color coordinates changing from white, (0.308, 0.317) at 299 K, to yellow, (0.416, 0.514) at 455 K. These results showed that these TLZ glasses are promising platforms for optical temperature sensing, for example, such as safety signal in high-temperature environment.

1. Jaque, D. and F. Vetrone., Nanoscale, 2012. 4(15): p. 4301-4326.
2. Vetrone, F., et al., ACS Nano, 2010. 4(6): p. 3254-3258.
3. Ximendes, E.C., et al., Adv. Functional Materials, 2017. 27(38): p. 1702249.

K-Energy Harvesting: Perovskite, Organic, Hybrid, Quantum Dots, and Dye-Sensitized Solar Cells

Advancements on double-junction silicon-perovskite photovoltaics

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New photovoltaic (PV) technologies need to be both high-efficiency and low-cost to reduce the generated energy cost per Watt. To achieve efficiencies above 29% at a reasonable cost, the most advanced and innovative research approach is to combine a silicon (c-Si) sub-cell with a low-cost wide bandgap absorbing material to form a monolithic tandem photovoltaic cell [1]. Tandem cells divide the solar spectrum into parts using multiple absorbing materials which allows them to collect photons more efficiently with reduced thermal effects. Perovskite materials have ideal properties for applications in tandem configurations, including high absorption coefficient, low sub-bandgap absorption and adjustable gap energies between 1.48 and 2.23 eV. Indeed, in April 2023 KAUST University has achieved a new world efficiency record for a silicon-perovskite tandem solar cell with a 33.2% certified efficiency [2]. In this talk, we will overview the most recent achievements on c-Si-perovskite integration covering aspects related to interconnection strategies, perovskite bandgap engineering, processing techniques, optical coupling, stability issues and up-scaling. We will also share preliminary results of the VERTICAL project, a cooperation between CTI and BYD Energy Brasil to develop high-efficiency optimized Si-perovskite tandem PV cells.

This work was supported by the grants #406558/2022-1 (CNPq) and #442553/2019-6 (SisNano/CNPq) and BYD Energy do Brasil (PADIS-Program).

[1] Fu, F., Li, J., Yang, T. C.-J., Liang, H. et. al. Adv. Mater. 34, 2106540, 2022.
[2] Best Research-Cell Efficiencies,
https://www.nrel.gov/pv/assets/pdfs/best-research-cell-efficiencies.pdf (accessed: April 17th 2023).

ORGANIC SOLAR CELLS (OSCs): MORPHOLOGY, LIGHT HARVESTING, EXCITON DISSOCIATION ANDENERGY TRANSFER STUDIES.

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The use of Organic semiconductors for photovoltaic devices presents advantages in processing and on the development of light and flexible devices, using techniques commonly used in the production of large areas, such as spray coating, roll to roll and jet printing ink. The nanostructure matters to efficient organic solar cells (OSCs) due to the compromise a bulk heterojunction (BHJ) formed by electron donor (D) and acceptor (A) components so that excitons created either at the D phase or A phase can efficiently quench at the D/A interface. For a system with optimized charge transport and extraction, the OSC efficiencyis essentially determined by the dissociation of those excitons at the heterojunction. In thiswork, some examples of these studies on the combination of organic materials in thin filmswill be presented in the fabrication of photovoltaic devices obtained by simple mixture in acommon solvent; generated by interfacial synthesi and by mini emulsion technique. Usingsimple mixture, it was possible to study devices from green solvent and in a ternarycomposition in order to rise the photon extracted number and consequently the powerconversion efficiency. The interfacial synthesis allows us to obtain new approach to nanostructure the films, thus obtain new optoelectronics properties and the mini emulsiontechnique allows organic semiconductor nanoparticles synthesis being dispersed in aqueousmedium offering the prospect of overcoming the use of chlorinated, inflammable and toxicsolvents from the manufacturing process of the devices. In addition, the use of commercialorganic solar cells will be presented and discussed in two projects installed at UFPR.

Synthesis and physical properties of low-dimensional0D, 1D, and 2D hybrid organic-inorganic halide perovskites

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Light-harvesting 3D organic-inorganic hybrid halide perovskites have been attracting considerable attention due to their excellent optoelectronic properties. Tuning in their electronic/crystal structures and photoelectric conductivity is at the heart of idealization of new materials, novel functionalities and/or improving the efficiency of optoelectronic devices. The important physical properties include suitable and adjustable band gaps, low exciton binding energy, large optical absorption coefficients, long hole-electron diffusion length leading to efficient charge separation/collection, and high defect tolerance. Compared with the conventional 3D perovskite, the low-dimensional family exhibits better environmental stability and more varied compositions, electronic, and crystal structures. In this talk, we will show some contributions of our research group on synthesis and physical properties of low dimensional 0D, 1D, and 2D organic-inorganic halide perovskites. In particular, we have grown low dimensional perovskites by using a recrystallization process starting from their 3D counterparts. The variation of dimensionality bringing about strong quantum confinement effect leads to change in physical/chemical properties which in turn offer a broad exploration research field for optoelectronic applications including light emitting and solar conversion devices.

Acknowledgment

The authors acknowledge the financial support from the Brazilian agency FAPESP 2021/11446-6, and CNPq 305601/2019-9. The authors are also grateful for the Multiuser Experimental Center of UFABC.

Advancing the stability of quantum dot-sensitized solar cells through CuS/carbon composite counter electrodes

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Quantum dot-sensitized solar cells (QDSSCs) are promising candidates among third-generation photovoltaic devices. Semiconductor quantum dots have several characteristics that have attracted much attention recently, including their adjustable bandgap, ease of processing at low temperature and high light absorption. An increase in energy conversion efficiency was reported for different types of QDSSCs in recent years, reaching in the 14% range, making them attractive for large-scale manufacturing. [1] The stability of QDSSCs, however, still remains inadequate for industrial application, especially when they are operated under sunlight and ambient atmosphere, being a barrier to be overcome for large-scale applications. Carbonaceous materials (carbon black, carbon nanotube, etc) have been widely applied as electrodes in solar cells, considerably improving these devices stability.[2] In this work, some of the degradation mechanisms of CuS and CuS/carbon composite counter electrodes (CE) used in QDSSCs were investigated. QDSSCs were prepared using CdS quantum dots and characterized through IxV curves and impedance spectroscopy, during 30 days. It was observed that an adequated control on the deposition and post-deposition parameters (concentration, temperature, time, etc), led to a decrease in some of the main factors responsible for the CE stability, such as an increase in the electrocatalytic activity and better adhesion to the substrate. The CuS/carbon composite CE resulted in a gain in the stability of QDSSCs that was related to the ability of carbonaceous materials to inhibit chemical photocorrosion of the electrocatalyst, which further indicates that CuS/carbon composite CE can become a potential electrode material in the field of solar cells.

Acknowledgments:
This work was supported by CAPES, CNPq, FAPEMIG and UFSJ.

Reference:
[1] A. S. Rasal, et al., Nano Energy, 94, 106854 (2022)
[2] A. S. Rasal, et al., Small, 18, 2202133 (2022)

Ag-Au bimetallic nanoparticles effect on perovskitesolar cells performance

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Electrical energy is fundamental for the economy, for science and for sustainable development. Therefore, it is important to seek more sustainable and efficient ways of producing and using electricity, in order to guarantee a safer and cleaner energy future for the next generations. Thus, Perovskite materials have excelled in energy conversion field being considered a potentially cost-effective alternative for the photovoltaic sector [1]. Inorganic perovskites CsPbBr3 have been considered of great interest since they are more thermally stable, due to the replacement of the organic cation by cesium in its structure. Solar cells made from this perovskite composition can be manufactured without the need for an inert atmosphere, presenting itself as a viable alternative when it comes to material greater stability [2]. Additionally, for perovskite solar cells, carbon electrodes are an interesting alternative to the traditional gold electrodes, thus increasing its useful life and reducing the cost of technology [1]. However these devices still suffer from unfavorable charge transfer process and limited light-gathering ability. Intending to boost photovoltaic performance, and evaluate the effect of surface plasmonic resonance, Ag-Au bimetallic nanoparticles (BMN) had been incorporated to devices. The Ag-Au BMN addition provides improvements in optical and morphological properties and improvement in photovoltaic performance, with regard to photocurrent (Jsc) response. In that way they can be used as an effective tool in the improvement of Jsc and consequently, with the possibility of the other parameters optimization, in the efficiency of photovoltaic devices.

Acknowledgements:

This work was supported financially by CAPES, CNPq, FACEPE. Also, the authors gratefully acknowledge to the Centro de Tecnologias Estratégicas do Nordeste (CETENE).

References:

[1] Bidikoudi, M., et al. Electronics 12.4 (2023): 992.

[2] Kumar, N., et al. Solar Energy 221 (2021): 197-205.

Amphiphilic main-chain poly(fullerene xylene)s

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Main-chain fullerene-based polymers are still relatively rare. However, because they combine the properties of fullerene, such as exceptional electron acceptance capability and semiconductivity, with those of polymers such as good solubility and excellent film-forming characteristics, they are gaining particular interest for their use as interlayers in photovoltaics and organic electronics. They exhibit many of the properties of fullerenes, combined with the ability to be formed as a regular thin film. For examples, recent work has demonstrated their use as interlayers in perovskite solar cells to give efficiencies around 23% and good stabilities[1], and that they might find use in ammonia sensors [2]. The methodology used to make these polymers can impact extremely heavily on its final physico-chemical properties. Therefore in this work, by way of introduction, we review the different known methods of preparing main-chain poly(fullerene)s and how those methods can modify the polymer's behaviour in application [3-6]. We further look to new methods for reacting with the chain-ends of these materials to expand their properties, especially by combining them with poly(ethylene glycol) to give amphiphilic materials. It is expected that these new materials might be of interest for 'green' processing of interlayers and drug delivery systems.

References

[1] J. Yin et al., Angew. Chem. Int. Ed. 2022, 61, e202210610.
[2] A. V. Santos Simõis et al., Materials Research, 2021, 24(1), 0435.
[3] M. Stephen, et al., Chem. Comm., 2016, 52, 61070.
[4] H. Santos Silva, et al. J. Polym. Sci., Pt B, Polym. Phys., 2019, 57, 1434.
[5] L. Roncaselli, et al. Physical Chemistry Chemical Physics, 2022, 24, 12442.
[6] R. C. Hiorns, et al. Macromolecules, 2009, 42, 3549.

CsPbBr3 QDs Changing the Morphology and Structureof MAPbI3 Films for Perovskite Solar Cells

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Perovskite solar cells (PSCs) are currently one of the most promising semiconductor materials for solar energy conversion, combining high efficiency, ease of manufacture, and economy. On the other hand, halide Perovskite Quantum Dots (PQDs) present intrinsic properties that may be interesting for photovoltaic applications, low defect density, compatibility with large-scale de-position techniques, and adjustable bandgap. In addition, all-inorganic perovskite has been shown to have higher stability than organic-inorganic hybrid perovskite (PK) materials under ambient conditions. An effective strategy to promote the long-term stability of PSCs has been coating moisture-resistant layers on the surface of the PK. Thus, PQDs are a promising candidate for interface engineering in PSCs due to their stability and adjustable gap position. Therefore, in this work, we investigated the influence of CsPbBr3 PQDs on the PSCs layer' s morphological, optical, and structural properties. The incorporation of PQDs in PK films was achieved by depositing its suspension onto the substrate surface and forming the MAPbI3 PK film by the sequential deposition method. Another approach was to deposit PbI2 followed by PQDs suspension and, finally, MAI; resulting in PK film with PQDs. Preliminary results of scanning electron microscopy show that the presence of PQDs leads to the formation of different morphology depending on the deposition procedure employed. Depositing PQDs before the PK formation resulted in films with nanocrystals with well-defined cube-shaped like, while the deposition of PQDs between PbI2 and MAI does not change the morphology of PK films. In addition, preliminary XRD results indicate that with the introduction of PQDs, the perovskite structure is kept. Further experiments using these films in PSCs are in progress.

Multimodal Spectroscopic Characterization ofPerovskites for Photovoltaic Applications

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Research in the last two decades has led to significant advances in the performance for Perovskite materials for photovoltaic (PV) applications [1]. Along with their significant advantages, PV Perovskites also tend to have strong vulnerabilities to environmental conditions, and are easily degraded from exposure to water, light, temperature and oxygen leading to poor long-term stability [2].

Some of the work being done today include designing Perovskite materials to have optimal optoelectronic properties for PV, uniform deposition of thin films, and proper protection or passivation of the resultant solar cells to enhance their long-term stability and performance. This work requires a variety of spectroscopic characterization tools which have historically been separate pieces of equipment. In this presentation, we show a novel multimodal spectroscopic characterization platform that combines a variety of spectroscopic modalities including Photoluminescence (PL), Raman, TRPL Electroluminescence for characterization PV Perovskites.

Figure 1: (a) Steady state Photoluminescence map of solar cells showing uniformity. Insert is a high spatial resolution map of a defect. (b) Time resolved Photoluminescence measurement of carrier lifetime away from, at the edge of, and within the defect shown in the insert in figure (a)

With this new instrumentation design and configuration, we demonstrate a vertically integrated platform for determination of several of the performance indicators of Perovskites for PV application. From basic material properties such as carrier dynamics all the way to PV efficiency in the resulting solar cells. Furthermore, these modalities can be readily added to any standard optical microscope.

Photoinduced Frenkel pair defect lead phase-segregation in halide perovskite alloys

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Alloys of halide perovskites can be very different from other tetrahedral semiconductors. III-V or II-VI semiconductors generally have good miscibility if the atomic sizes are similar, andtend to phase-separate below a given miscibility temperature if their atomic sizes are sufficiently mismatched. Alloying two HPs with different halogen ions X and X’ can lead to their complete miscibility in the dark, and phase-segregate into their parent compounds under brief light exposure. This effect has been observed in hybrid and inorganic alloys of HP. Thermodynamic properties or intrinsic defects were invoked to explain the phase segregation under light or remixing under dark in alloys of HP, however, these models do not bring a complete explanation for the process. In this work, we clarify via first principles defect calculations that a Frenkel like pair (FP) defect complex is the main agent for phase segregation. This process can be described as a sequence of steps: First, the photogenerated holes, delocalized at the valence band, can lower their energy when I dislodge from its lattice site, leaving behind an iodine vacancy (V_I) and forming a FP complex $(V_I+I_i)^{2+}$. The associated FP tends to form on alloys with an intermediate concentration of I and Br, and in a I-rich local chemical environment. Second, in the same intermediate alloy concentration, the FP tends to dissociate into $V_I^{1+}+I_i^{1+}$. Third, the migration of I into $V_I$is faster and can produce I-rich and I-depleted regions. The phase segregation process stops when the local chemical environment of alloys becomes like the separated parents’ composition, where the FP does not form. In the dark and under thermal annealing, the excess of holes is suppressed, and the entropy increases in the halide perovskite killing the photoinduced $V_I$and $I_i$and returning to the original form.

Acknowledgement
FPS and GMD: FAPESP (grants 2021/11786-1, 2019/21656-8). AZ and FPS: U.S. Department of Energy’s Award Number DE-EE0009615.

Stability study on Perovskite-based solar cell deviceswith salophen

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In recent years, hybrid perovskites have been developed with an organic and polar structure (with a permanent electric dipole) CH3NH3. In these materials, the inorganic part is a lead halide (PbI3, PbBr3 or PbCl3). The photovoltaic performance of these perovskites is due to dipole fluctuations of the organic part CH3NH3. This research shows that it is possible to improve the stability of perovskite MAPbI3 by properly incorporating salophen atoms into its matrix; thus, the results indicate that perovskite MAPbI3/salophen has a better stability. This organic ligand can coordinate with the Pb (II) metal centre in tridentate or tetradentate form through a combination of any nitrogen or oxygen donor atom. It can also be in the mono-deprotonated or neutral form. Consequently, it is sterically ideal for establishing tetryl bonding interactions with electron-rich nitrogen our bromide atoms. The improvement is linked to the structural transition from the tetragonal to the cubic phase, a more stable structure as shown by the X-ray diffraction (XRD) results. In this research, we performed the preparation of the perovskite precursor solution, the deposition of films by spin-coating, and characterisation to study the structure and photophysical behaviour of MAPbI3/salophen. The perovskites were deposited on transparent conductive fluorine-doped tin oxide (FTO) substrates, the films were characterised by X-ray diffraction (XRD), UV-vis absorption, and also emission (PL) and excitation (PLE) photoluminescence. Perovskite- based solar cell devices containing salophen with 18.22% efficiency, given by the PCE index(Power Conversion Efficiency). The research also reinforces the study of the stability of theoptical and electronic properties of this perovskite; in order to understand the mechanismresponsible for the degradation of this material, this point is essential for us to build materials with better stability and photovoltaic efficiency.

Synthesis and Characterization of Stable 2D Perovskite Incorporating a Thieno-Pyrrole-Containing Ligand and 2D/3D Interface Engineering

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Two-dimensional (2D) perovskite materials have been attracting increasing attention due to their higher stability in ambient conditions compared to 3D perovskite materials, resulting in increased stability of perovskite optoelectronic devices. Furthermore, chemical functionalization of organic spacers is an effective approach for tuning the 2D Ruddlesden-Popper (RP) electronic properties. Here, we report the synthesis and characterization of a new 2D layered perovskite incorporating a π-conjugated thieno-pyrrole ligand $(TP\text{-}TEA)_2PbI_4$. Structural and morphological analysis confirm the formation of microplatelets 2D RP perovskites with triclinic symmetry. The hydrophobic organic spacer layer provides natural protective barrier for transport of ions and moisture, offering significantly improvedlong-term stability for these 2D perovskites. We have systematically investigated the morphological, structural, optical, and electronic properties of these 2D perovskite separately and placed them on the top of 3D films, forming 2D/3D heterostructures for studyin perovskite solar cells. The absorption of 2D/3D perovskite films showed similar bandgapand absorption profiles in comparison with that of control 3D perovskite films. Time- resolved photoluminescence shows that the isolated new 2D RP perovskite has longer charge carrier lifetime when compared with PEA and TEA-based perovskites. In addition, the recombination rates for TiO_2/perovskite are faster when using the new ligand in 2D/3Dheterostructure, indicating more efficient interface charge transfer at the hole-transport layer/perovskite interface. These results are discussed based on the passivation effect brought by the new ligand which increased the overall stability of the perovskite layer anddecreased the nonradiative recombination rates.

Acknowledgements

The authors acknowledge the financial support from the Brazilian agency FAPESP under grants 2021/11446-6, 2018/14181-0, and 2021/10585-2.

Synthesis of perovskite thin films from sputtered lead iodide

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Synthesis of high-quality organolead trihalide perovskite ($CH_3NH_3PbI_3$) has been the subject of intense investigation due to their attractive optical and electronic properties, e.g., direct bandgap, high absorption coefficient, and ambipolar charge transport. The main route used to synthesize $CH_3NH_3PbI_3$ thin films for highly efficient solar cells is based on spin-coating deposition. This approach has produced solar cells with power conversion efficiencies higher than 25%. In this work, Lead Iodide (PbI_2) thin films, were deposited through rf- sputtering and then successfully converted into perovskite films using a two-step process. The sputtered PbI_2 films were evenly deposited on large areas and on different substrates. The as-deposited PbI_2 thin films were converted into $CH_3NH_3PbI_3$ by immersion in a methylammonium iodide (CH_3NH_3I) solution. By this approach, we synthesized $CH_3NH_3PbI_3$ thin films with good optical and structural properties. The perovskite films presented an optical bandgap of 1.5 eV and crystalline structure in accordance with the reported in literature. This new method is fully compatible with large-scale manufacturing. Besides, it has the potential to open a way toward reproducible and high-efficiency $CH_3NH_3PbI_3$ solar cells.

Acknowledgments

CNPq and FAPESP.

References:

[1] Silva Filho, J. M. C., Ermakov, V. A., Marques, F. C., Scientific Reports, 8, 1563 (2018)

[2] Silva Filho, J. M. C. et al., Crystal Growth & Design, 20, 1531 (2020)

Thermal degradation kinetics of halide perovskites with mixed organic cations

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Halide perovskites are a group of materials extensively studied in the last decade due to their versatility of compositions and properties that allow wide optoelectronic applications. This potential, however, is limited by the low stability of these materials and their devices. Many degradation routes are known, among which thermal degradation is of particular relevance in compositions containing organic cations. Although abundant literature has been devoted to this topic, little attention has been given to the effect of different cations onthe kinetics of degradation in these halide perovskites. In the present work, we studied the thermal degradation kinetics of perovskites of compositions MAPbI3, FA0.10MA0.90PbI3, GA0.10MA0.90PbI3 and GA0.05FA0.05MA0.90PbI3, where MA, FA and GA are respectively the organic cations methylammonium, formamidium and guanidinium. The kinetic constants obtained by changing X-ray diffraction patterns as a function of time in samples subjected to temperatures between 150 and 200°C reveal that the compositions FA0.10MA0.90PbI3 e GA0.05FA0.05MA0.90PbI3 show slower thermal degradation than MAPbI3, where the GA0.05FA0.05MA0.90PbI3 composition appears to be the most stable. However, contrary to general expectations, the composition GA0.10MA0.90PbI3 has higher kinetic constants than MAPbI3. Furthermore, the composition GA0.10MA0.90PbI3 shows a degradation route different from the others. This work highlights the importance of the rational choice of the different organic cations to be used in a halide perovskite composition for a given application of interest.

Ti_3C_2 MXene as efficient additive for high-performance inverted perovskite solar cells

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Inverted perovskite solar cells (IPSCs) have drawn attention due to their low-temperature fabrication process and negligible hysteresis. [1]. Even though showing several advantages, these inverted (p-i-n) structures still lack of desirable high performance and long-term stability. MXenes, an emerging class of two-dimensional (2D) transition metal carbides and nitrides, has been studied due to their unique properties such as high electrical conductivity, high transmittance and high hydrophilicity [2]. In this work, high performance IPSCs were obtained by inserting Ti_3C_2MXene as passivation agent in the perovskite layer. Low concentration of $Ti_3C_2T_x$with respect to $Cs_{0.17}FA_{0.83}Pb(I_{0.83}Br_{0.17})_3$PVK resulted in increase of $J_{sc}$and FF. Moreover, V_{oc}improved to 1.12 ± 0.01 against only 1.08 ± 0.03 V of the pristine devices. Then, the improvement of the functional parameters of PSCs contributed to substantially increase PCE to 21.27 ± 0.66 % when compared to the pristine devices (18.80 ± 1.43 %). Photo-CELIV characterization showed improved charge mobility and higher density of extracted electrons of PSCs after addition of MXenes. Fitting of TPV curves showed longer charge recombination time of the devices containing MXenes. These results were accompanied by a decrease of the charge transfer resistance and an enhanced charge recombination resistance, as shown by EIS. These features can be mainly attributed to the high electrical conductivity of MXene, which works as an effective charge transfer path and prevents their recombination at the perovskite/ETL interface. This premise was confirmed by the reduced number of calculated traps of PVK-Ti_3C_2, indicating a defect passivation by the MXene.

Acknowledgments:
To FAPESP (Grants: 20/16470-0, 20/12356-8, 22/09093-0, 22/08848-8), UNESP (PD: 4223) and FINEP (Grant: 01.22.0289.00 (0034/21)) for the financial support.

References:
[1] X. Lin et al, Energy Environ. Sci., 3823 (2020)
[2] A. Yakusheva et al., Small, 2201730 (2022).

Ab-initio phase diagram of CsPbBr3

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Lead halide perovskites are promising for use in solar cells and optoelectronic devices, but stability concerns limit their possible applications. Understanding their atomic structure is important for the optimization of performance in photovoltaics, radiation detection and thermoelectric systems. Cesium lead halides (CsPbX3) are particularly promising due to their stability at high temperatures and desirable properties such as band gaps. However,CsPbX3 are temperature-sensitive and exhibits different phases at different temperatures. This theoretical study aimed to analyze the variation of the structure of CsPbBr3 perovskiteas a function of temperature. To achieve this, Ab Initio Molecular Dynamics were performedin an NPT ensemble with a completely flexible cell, keeping the pressure and temperature constant. Multiple temperature values between 150 and 500 K were considered, while the pressure was kept at zero in all simulations. The calculations were performed at the DFT level using the CP2K software. The variation of the average volume of the CsPbBr3 unit cell at different temperatures was measured and the corresponding average structures were determined. In addition, the behavior of the structural parameters of the cells in each simulation was analyzed, allowing to detect that the average structure is not present all the times during the simulation, but the system shows different geometries at a given temperature. The frequency with which these different geometries were observed in the simulations could be calculated. Angle tilt and band gap analyses were also carried out. This study has allowed us to conclude that the geometry of the CsPbBr3 perovskite undergoes fluctuations at specific temperatures. In addition, by analyzing the tilt angle of PbBr6, the mechanisms by which phase changes occur in the perovskite structure as a function of temperature have been elucidated. This work has been funded by FAPESP and by CNPq, through the Materials Informatics INCT.

γ-CsPbI3 nanoparticles: light-induced self-assembly and optical properties

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Metallic halide perovskites (MHPs) have excellent optical properties, which has led to awake the interest of the scientific community, and their implementation in a variety of optoelectronic devices, such as solar cells, light-emitting diodes, lasers, etc. However, a deep understanding of the fundamental properties of this kind of materials is still needed. Inthis work, we synthed γ-CsPbI3 nanoparticles capped with (3-Aminopropyl)triethoxysilan (APTES) and oleylamine by the hot injection method. The nanoparticles were cubic of 32 nmin . We irradiated the nanoparticles with a solar simulator, and we could observe that thatthe nanoparticles self-assembled into nanorods. Moreover, we studied the optical properties obtained from the photoluminiscence spectra at low temperature. Our results showed that the self-assembly process does not affect considerably the interaction of excitons and phonons, but it could affect negatively the crystalline network quality. Besides, we observed that the detachment by light of the capping agent could be responsible for the self-assembly. The fundamental processes that are responsible for this effect are still unknown, and further studies will be focused on the study of the factors that affect the final characteristics of the assembled nanorods.

Acknowledgements: Programa de Desarrollo de las Ciencias Básicas (PEDECIBA), Laboratorio de Alta Resolución, Udelar.

Biopolymer membranes modified with graphene oxide and PEDOT:PSS as flexible substrates for photovoltaicdevices

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The great challenge for photovoltaics is to obtain high efficiency and low-cost devices.Nanomaterials allow the improvement of electrical conductivity and light absorption.Graphene oxide (GO),with high surface/volume ratio,great mechanical and chemical stability and high electric and thermal conductivity[1] is promising nanomaterial in this field.Otherwise,Poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate)(PEDOT:PSS),as a transparent and stable polymer,with high conductivity and ease of processing,is widely used in organic solar cells [2].So,a combination between PEDOT:PSS and GO can improve,concurrently,electrical conductivity and mechanical properties of biopolymer membranes.Biopolymers are interesting options for photovoltaic substrates,because of low-cost production,versatility and low environmental impact.Xanthan is a biopolymer already used in electronic devices[3] and,with the addition of plasticizers and crosslinkerrs,forms transparent and flexible substrates.The purpose of this study was prepare xanthan biopolymeric substrates modified with GO and PEDOT:PSS to obtain optimized electrical and optical properties for organic photovoltaic devices.For the preparation of biopolymeric membranes,xanthan was dissolved in deionized water,ethylene glycol was used as a plasticizer,and glutaraldehyde was used as a crosslinker.GO was added in a solution with different concentrations of it and PEDOT:PSS was added during membrane synthesis or after,by spin coating deposition.Morphological,electrical and optical properties have been studied and correlated with the preparation methods evaluated in this study.Preliminary results indicated a increase in the electrical conductivity and stability overtime for membranes modified withGO or PEDOT:PSS.Acknowledgements:CAPES,FAPERJ.References:[1] J.E.D Vieira et al.Revista Eletrônica de Materiais e Processos,v.11,n.2,p.54-57,2016 [2] S. Rafique et al.Sci.Rep.7 (2017)1-10 [3] F.C. Tavares et al.J.Appl.Polym.Sci. 5976 (2018)

Characterization of Carbon-Based Electrodes for Application in Third-Generation Solar Cells

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Abstract: The increase in energy demand, environmental problems and the possible scarcity of non-renewable energy sources are some of humanity''s recent concerns. In this scenario, scientists are looking for ways to develop technologies for energy production from renewable sources and with low environmental impact. Third-generation solar cells are a possible solution for sustainable and low-cost energy generation [1]. However, one of the challenges of this generation is to improve stability and reduce energy losses. Carbon-based electrodes have been widely studied, seeking to increase the lifetime and energy conversion efficiency of 3rd generation cells, due to their electrical and mechanical properties, stability, low cost and high surface area [2]. In this work, carbon electrodes were prepared from carbon-based pastes (Multi-Walled Carbon Nanotube (MWCNT) and Carbon Black) of different compositions, in order to investigate their electrical properties. The electrodes were characterized by Cyclic Voltammetry and Polarization Curves techniques. Electrical conductivities were obtained for the different compositions of the electrodes used. Cyclic voltammetry allowed investigating the stability of the electrodes as a function of concentration, allowing an optimization of the concentrations of the paste components. It was concluded that the carbon paste based on MWCNT, presented better conductivity, being more suitable for application in solar cells. In the future, we seek to understand the effect of the morphology of these electrodes (roughness, surface area, thickness) applicated to 3rd generation solar cells.

Acknowledgments:
We thank UFSJ for the project opportunity. This work was supported by CNPq, CAPES and FAPEMIG.

References:

[1] Akash S. Rasal, et al.,Stability of quantum dot-sensitized solar cells: A review and prospects,Nano Energy,Volume 94,2022.

[2] Wu, M., et al. Carbon Counter Electrodes in Dye-Sensitized and Perovskite Solar Cells. Adv. Funct. Mater. 2020, 30.

Copper Delafossites as Hole Transport Material for Efficient and Stable Inverted Perovskite Solar Cells

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Hybrid perovskite-based solar cells have been widely studied throughout thelast decade since it allies the inorganic high conductivity and performance to theorganic processabilityand low cost. The current hindrance to the large-scaleproduction of this kind of solar cell isits stability facing degradation processes toadjacent layers and external factors, mainly to humidity. P-type metal oxidesemiconductors are promising candidates as a hole transport material ininverted perovskite solar cells, due to their enhanced optoelectronic properties:improved hole mobility, suitable bandgaps and high transparency, and fittingenergy levels with the active layer [1]. These materials also provide chemicalstability and a humidity barrier to the active layer. The present study involvesthe experimental characterization of copper delafossite thin films as analternative to the hole transport material conventionally used. Copper aluminatedelafossite thin films have been deposited using reactive RF-sputtering andcharacterized by X-ray diffraction, Raman Spectroscopy, X-ray PhotoelectronSpectroscopy, Four-probe Resistivity measurement, and UV-Vis Spectroscopy. Scanning Electron Microscopy, Contact Angle measurements and Ellipsometrywere also performed to study the deposition dynamics and the thin film surface.

Acknowledgements:

CINE - Shell (ANP) / FAPESP grant n. 2017/11937-4, 2018/04596-9 and2018/04595-2

References:

[1] Akin, S., et al. Inorganic CuFeO2 Delafossite Nanoparticles as Effective HoleTransport Material for Highly Efficient and Long-Term Stable Perovskite SolarCells. ACS Applied Materials Interfaces, 11, 48, 45142-45149, 2019.

$Cs_3Sb_2X_9$ perovskite synthesis using design ofexperiments

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In the past decade, lead halide perovskites have garnered significant attention due to their impressive photoluminescent properties and photoconversion efficiencies. However, their application has been hindered by their instability towards heat and moisture, which results in the leaching of lead into the environment. In recent years, full-inorganic lead-free perovskites have emerged as a promising alternative, with tin being the most common substitute. However, the oxidation of Sn^{2+}poses a significant challenge, making its application difficult [1]. To overcome this challenge, researchers have turned to antimony halide perovskites, which possess promising luminescent properties [2,3]. In this work we synthesized antimony halide perovskites $Cs_3Sb_2X_9$(X = Cl, I) through a simple precipitation process [3]; in this method, $SbX_3$is dissolved in acid media and adding the corresponding cesium halide to the solution achieves instantaneous perovskite precipitation. To optimize synthetic conditions and obtain a higher yield, we employed a three-factorial design of experiments (2^k, k =3), with temperature, solvent, and precursor concentration being the factors studied. To our knowledge, there are few studies regarding synthetic conditions to these materials so our work may be of great value to the field.

Acknowledgements: CNPQ and Marcos José Leite Santos.

References:
[1] Fan B. et al., Solar Energy Materials and Solar Cells, vol. 184, 15-21 (2018)
[2] Zhang, J. et al., ACS Nano, vol. 11, 9294-9302 (2017)
[3] Wang, X., et al., Inorganic Chemistry, vol. 59, 15289-15294 (2020)

Degradation Study in Organic-Inorganic Perovskite Halides Thin Films on Different Substrates

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In this work, perovskite thin films of organic-inorganic multication lead halides (HOIP) were studied to understand how the degradation process affects the optoelectronic properties of the films on different substrates. Perovskite films were synthesised using the one-step method, which consists of dissolving the reagents in well-defined stoichiometric amounts utilising a combination of solvents that produces a precursor solution. Then, the solution was deposited on the substrates by the centrifugation technique and, before the end of the centrifugation process, an antisolvent was dripped. Then, the substrate containing the precursor film was placed on a heated plate at a temperature of 120°C for 30 min for perovskite formation to occur. Glass substrates, fluorine-doped tin oxide (FTO), indium tin oxide (ITO) and poly-3,4-ethylene dioxythiophene:poly(styrene sulfonate) (PEDOT: PSS) were used for the deposition. Characterize the films, traditional spectroscopy techniques were used to characterize materials with optical properties, such as UV-Vis absorption and Photoluminescence (PL), and X-Ray Diffraction (XRD) techniques will be used to describe the film structures. These techniques have allowed a systematic investigation of the optical properties of films and the monitoring of how they change as the material degrades due to exposure to heat and humidity. For studies of the degradation process, the degradation protocol was carried out over 30 days, in which day 1 was the specific day in which the films were synthesised and during the first five days the optical characterization measurements were taken. Once a day at the same time, and for the remaining 30 days, optical measurements were retaken on days 10, 20 and 30. As for the structural characterisation measure, a second batch of films was synthesised, which measurements were taken on the 1st, 5th, 10th, 20th and 30th.

Acknowledgements: CAPES, CNP q, Finep, Lamuta.

Deposition and Characterization of Cobalt Oxide by RF Magnetron Sputtering to apply as Hole Transport Layerin Perovskite Solar Cell.

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Nowadays, perovskite solar cells (PSC) draw great interest in the field of energy harvesting due to rapid increase in the efficiency, reaching above 25%, and as a substitute for the silicon solar cells in the near future [1]. Nevertheless, not the only, but one of the problems of these devices is the stability, addressed to different causes [1]. Usually, those cells are made using the perovskite layer as an absorb material, between selective transport layers, being called p-i-n for the case that a hole transport layer (HTL) comes beforehand the perovskite layer. The correct selection of materials for selective layers is essential for good performance of the device. Thus, the use of metal oxide layers has been investigated in order to improve the stability and make it commercially viable. In this way, the cobalt oxide (CoO) is presented as a potential option [2]. Therefore, this work explored the deposition of CoO by reactive sputtering and its use as a HTL in the PSC. Sputtering is a well-known method to deposit metal oxides, and tuning the deposition parameters allows the change of the film properties [3], which can lead to desirable conditions to apply the CoO as HTL in the devices. The devices manufactured with the CoO as HTL, reached 6 % of efficiency. Although, the efficiency may be beneath the expected, this gives insights in ways to improve the layer deposition and its whole in the device, concerning the comprehension of the causes that limit the performance of the cells.

Acknowledgements
CAPES - PROEX Scholarship, CAPES-PrInt Process No: 88887.571101/2020-00, FAPESP Process No: 2020/12356-8.

References
[1] Kim, J.Y., et al. Chemical Reviews. 120 (15). 2020.
[2] Azevedo Neto, N.F., et al. Journal of Vacuum Science Technology A: Vacuum, surfaces, and films. 36 (6). 2018.
[3] Shalan, A.E. et al. ACS applied materials interfaces. 8 (49). 2016.

DETERMINATION OF PARAMETERS FOR ELECTROSPINNING OF POLY(METHYL METHACRYLATE)

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This work aimed to study the parameters for obtaining PMMA nanofibers to use as the shell of a coaxial nanofiber structure. These structures will be used as the active layer of a BHJ solar cell, the core is a conductive polymer, and the shell is PMMA. The first parameter studied was the type of solvent with acetone, chlorobenzene, and dimethylformamide. The polymer has good solubility in the three solvents, and acetone was chosen because it has a lower boiling temperature value. The second parameter was the concentration of the PMMA solution in acetone, and concentrations of 5%, 7.5%, and 10.0% by mass were tested. For this study, a viscosimetry analysis of the solution was applied. The samples used for electrospinning were those with 5% and 7.5% concentrations since the 10.0% solution presented a very high viscosity value concerning the other solutions. The studied electrospinning parameters were the distance between the needle and the collector, the application of the electric field, and the pump flow. The distance was varied by 10.0 cm, 12.5 cm, and 15.0 cm. The applied electric field values were 15 KV, 17 KV, and 20 KV. The pump''s flow changed to 0.5 ml/h, 1.0 ml/h, and 2.0 ml/h. The most promising set of samples, which resulted in the best PMMA nanofibers, without beads and in the suitable thickness for the application, was the one with the parameters of 5% concentration by mass, applied voltage of 20 KV, flow rate of 0.5 ml/h and distance between needle and collector in values of 7.5 cm, 10.0 cm, and 12.5 cm.Acknowledgments:Coordenação de Aperfeiçoamento de Pessoal de Nível Superior, CAPES Foundation

DEVELOPMENT OF SOLAR CONCENTRATOR BASEDON LUMINESCENT NANOCRYSTALS

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Luminescent Solar Concentrators (LSC) were introduced nearly four decades ago as a potential complement to silicon solar cells and are considered a powerful photonic platform.1 They consist of a semitransparent glass or polymer waveguide doped or coated with highly emissive luminophores, which after absorbing sunlight re-emit photons at longer wavelengths. Total internal reflection guides the luminescence to the edges of the waveguide, where it is converted into electricity by photovoltaic cells coupled to the concentrators. This form of light concentration is passive and inexpensive, without the need to using expensive and bulky equipment. In addition, the inherent ability to indirectly illuminate connected photovoltaic cells gives the LSC device the added benefit of being less sensitive to shading than conventional solar cells.2 In this work, the development of a solar concentrator at based on Dimethylpolysiloxane (PDMS) polymer, doped with quaternary quantum dots (QDs) of CuZnInS/ZnS, coupled to a $CuInS_2$solar cell was described. Due to the suppressed reabsorption and the high emission efficiencies of the quantum dots synthed, an efficiency increase in the solar cells was achieved compared to the ones where the solar concentrator was not used. In this way, it was possible to demonstrate the use of stable and ecologically correct QDs to produce large-area LSCs, capable of increasing the optical efficiencies of solar cells.

Acknowledgments:

This work was supported by CNPq, FINEP, CAPES and FAPEMIG.

References:

1. Papakonstantinou, I.; Portnoi, M.; Debije, M. G. Adv. Energy Mater. 2021, 11, 1.
2. Moraitis, P.; Schropp, R. E. I.; van Sark, W. G. J. H. M. Opt. Mater. (Amst). 2018, 84, 636.

Dipole Configuration Importance of the OrganicSpacers and Cations Interactions in Quasi-2D Perovskites: an *Ab Initio* Study

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$BA_2MA_{n-1}Pb_nI_{3n+1}$ are promising 2D Metal Halide Perovskites (MHPs) that have been largely investigated in the last few years and are emerging as an alternative to their former 3D $MAPbI_3$counterpart because of their attractive structural versatility and tunability of optical properties for solar cells. This 2D MHPs are based on methylammonium ($MA = CH_3NH_3^+$) cation and the hydrophobic butylammonium ($BA = CH_3(CH2)_3NH_3^+$) organic spacer, which promotes more moisture stability. A theoretical approach capable of describing a correlation between local distortion and optoelectronic properties, such as gap energy and absorption coefficient, is challenging. Here, we analyze via ***ab initio***density functional theory how the structural and electronic properties of the Pb_nI_{3n+1}octahedrons behave under partially canceled dipole moments through the NH_3^+terminal cations from MA and BA [1] - no excitonic properties are included. It is well-known that at room temperature, the MA cation begins to rotate inside the cage. However, our discussions focus on dipole moment orientation at 0 K as a vital step to advance the knowledge about 2D MHPs. For n = 1, 2, 3, 4, and 5 layers, employing a relativistic treatment with the DFT-1/2 quasiparticle and spin-orbit coupling (SOC) corrections, our results show that the octahedrons surface via no dipole moment cancellation induces the best data calculated and is in good agreement with experimental gap energies. In the opposite scenario, the dipole moment cancellation promotes the closing of the gap energies and shows an electron depletion mechanism. Furthermore, the interaction octahedron-MA yields high metal off-centering associated with strong SOC which resulted in bulk Rashba splitting. Finally, only the no dipole moment cancellation scenario converges to the bulk absorption coefficient and can keep the performance of 2D perovskites comparable to their 3D counterparts.

References:

[1] Diego Guedes-Sobrinho ***et al.***, Sci. Rep. 13, 4446 (2023)

Does GO in the buffer layer influence the opticalproperties of P3HT films?

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In this work, the films were deposited using the spin-coating technique based on the variation of the active layer, which may be the Poly- (3-hexylthiophene) polymer (P3HT) or the P3HT:PCBM blend, and the buffer layer, which may be the PEDOT: PSS blend or the PEDOT: PSS: GO blend, and after deposition, optical and morphological characterizations were performed to study the photophysical properties and the interaction between the different deposited layers. The films were deposited on a glass substrate with an Indium tin oxide (ITO) layer. Afterwards, optical characterizations were performed using Ultraviolet-Visible Absorption Spectroscopy (UV-Vis), Photoluminescence Spectroscopy (PL), Photoluminescence Excitation Spectroscopy (PLE), and Emission Ellipsometry (EE), as well as morphological characterization using Atomic Force Microscopy (AFM), which was also used to measure the thickness of the films. As a standard deposition method, the rotation speed was set at 3000 RPM for the buffer layer and 2400 RPM for the active layer, thus comparing the variations in optical measurements according to the films so that the thicknesses are similar. It was noted that GO has excellent influences on optical measurements and the interaction it presents with PCBM. Both gave its quenching effect, as observed in the PL measurement and made the transfer of energy more uniform concerning the film ITO/PEDOT: PSS/P3HT, as seen in the EE measurements. However, this work helps the developers understand the electronic interactions between the polymers, thus optimizing the photovoltaic devices since the GO in the buffer layer influenced the photophysics of the samples.

Effect of hysteresis on electrical properties in hybrid perovskite

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The use of Perovskites for the development of solar cells proves to be a good option due to the large absorption gap and has conversion efficiency approaching 25%. Lead triiodide methylammonium halide (MAPI) is a hybrid perovskite often applied in photovoltaic devices due to the low cost of reagents and fast manufacture. We noticed that hysteresis is frequent in perovskite, and it is a problem to get the actual efficiency of perovskite. We built a capacitor-like device, where the perovskite is in direct contact with the metallic layer. The perovskite has a manipulated deposition region, allowing more control of the device and enabling similar reproductions, using the two-step spin coating method. To speculate on the origin of hysteresis, we first understand if it originates from the transport layers, from the perovskite or even from the interface contacts [1], the effect of passivation changes the transient time scale of capacitive current. We used Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) to analyze layer thicknesses. Using the four-point probe measurement technique, a forward and backward J-V sweep was performed, trying to identify the hysteresis behavior. We seek to understand if the origin of hysteresis occurs due to a possible ion migration, velocity of transient capacitive current or other factors [2] to develop solutions for the hysteresis and for further progress to improve the efficiency of devices. The hybrid perovskite CH3NH3PbI3 shows a capacitive effect in the J-V measurement, which implies an intrinsic ferroelectric behavior like the classical ceramic perovskite. We use theoretical models to realize computer simulations and complement the understanding of electronic carrier transport.

References:

[1] SNAITH, H. J. The journal of physical chemistry letters, v. 5, n. 9, p. 1511-1515, (2014).
[2] CHEN, B. The journal of physical chemistry letters, v. 7, n. 5, p. 905-917, (2016).

Impact of MoS_2 on the active layer for high-performance p-i-n perovskite solar cells

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The perovskite active layer (PVK) plays a critical role in the performance and the stability of perovskite solar cells (PSCs). An alternative to improve the performance of the PSCs, the incorporation of additives in the PVK layer during the preparation of the precursor's solution has drawn attention [1]. Recently, two-dimensional transition-metal dichalcogenides (TMDs), such as MoS_2, $MoSe_2$and $MoTe_2$, have been explored in the different layers of PSCs due to their admirable photostability, quantum efficiency and tunable band gap [2]. In this context, we explore the impact of MoS_2 -doped $Cs_{0.17}FA_{0.83}Pb(I_{0.83}Br_{0.17})_3$active layers in inverted PSCs. The incorporation of MoS_2into the PVK was confirmed by XDR patterns. Moreover, the XRD patterns showed an increase of crystallinity and a suppression of PbI_2formation with the increase of MoS_2content. High performance devices were obtained by the MoS_2-doped the active perovskite layer. The addition of the MoS_2resulted in a considerable increase of J_{sc}, $V_{oc}$and FF. The improvement of these parameters contributed to a substantial increase of PCE (22.4%) when compared to pristine active layer (19.24%) based devices. Photo-CELIV characterization showed improved charge mobility and higher density of extracted electrons of PSCs with the incorporation of MoS_2. An increase of charge recombination time with addition of MoS_2were shown in transient photovoltage measurements. In addition, the EIS measurements showed a decrease of the charge transfer resistance and an enhanced charge recombination resistance with addition of MoS_2.

Acknowledgments:
To UNESP (Grant: 4224), FAPESP (Grants: 20/16470-0 and 20/12356-8), FINEP (Grant: 01.22.0289.00(0034/21)) for the financial support. To Multiuser Experimental Center (CEM) from UFABC.

References:
[1] Z. Guo et al, Small, 14, 1802738 (2018).
[2] D. Vikraman et al. Small, 18, 2104216 (2022).

Improved photoabsorption with blocking layer optimization in DSSC cells

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The reduce pollution and harmful gas emissions in the atmosphere, implementation of solar panels is one of the solutions, currently being one of the most exploited renewable energy sources and with excellent results. In the search for photoabsorbent materials with high performance and lower production costs, the route via microwave-assisted hydrothermal (MAH) has been used [1]. A blocking layer and the photoactive material of the solar cell characterize it as third-generation associated with hybrid-type perovskite. The main objective of this work is to explore materials synthed by the MAH method to improve photoabsorption when applied in solar cells. The initial tests are being carried out with the variation of the photoabsorber material: TiO_2, $KNbO_3$ and CaF_2 doped with rare earth ions. The deposition of the material was carried out using the spin-coating technique, by which some deposition parameters were varied, such as rotation and number of layers. Through the *JXV* curve, the partial results obtained show that $KNbO_3$ is a promising material for the blocking layer and can replace the usual TiO_2. Among all the cells made, the materials that have *Eu* and *Dy* ions had the best photocurrent results, attributed to the increase in the absorption band associated with the characteristic transitions of the dopant. Optical and morphological characterizations were also performed to investigate the influence of the deposition parameters used to assemble the solar cells.

Acknowledgements: This research used resources from the laboratory CCAF - UFPel and LPCM from the Federal University of Sergipe - SE.

References:

[1] LILGE, Tatiane Strelow et al. Dalton Transactions, v. 49, n. 25, pág. 8540-8548, 2020.

Improvement of ZnO/ZnS photoelectrodes through the addition of graphene oxide

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Brazil stands out for having a predominantly renewable electrical matrix. In 2023, photovoltaic energy became the second largest source of renewable energy in Brazil, indicating a growth of 84% in less than a year. In this study, graphene oxide will be introduced to the ZnO/ZnS compound to form the photoelectrode of the dye-sensitized solar cell, aiming to reduce the electronic mobility problem of ZnO [1]. Moreover, it is expected that the graphene will enhance the anchorage of ZnS particles on ZnO, providing a better solar absorption. In this context, the purpose of this study is to understand how the insertion of graphene in different amounts in the ZnO/ZnS compound affect the system, evaluating the influence on the power conversion efficiency. To realize the production of solar cells, initially the ZnO/ZnS + GO will be synthed using the microwave-assisted hydrothermal method, which offers a shorter synthesis time and greater homogeneity. To the ZnO synthesis, sodium hydroxide and zinc acetate will be used as precursors, for the ZnS coating, will be used zinc chloride and thioacetamide because they presented better results in studies performed by our research group [2]. The insertion of graphene into the system will happen at the time of ZnS coating, to evaluate how it will influence the system under the presence of electromagnetic waves. Subsequently, a dye-sensitized solar cell will be assembled, where the photoelectrode will be a film formed by ZnO/ZnS+GO. To evaluate the results, x-ray diffraction will be performed to verify the crystalline phase, Scanning Electron Microscopy to verify the and distribution of the crystals, and photovoltaics measurements to obtain the power conversion efficiency.

Acknowledgements:
CNPQ, Capes, Fapergs, UFPel, Unipampa and CCAF.

References:
[1] Fernandes CD, Ferrer MM, et al., New Journal of Chemistry 47., 20600-9 (2020)
[2] Farjadian F, Abbaspour, et al., ChemistrySelect 5., 10200-19 (2020)

INCORPORATION OF Cu-Zn-In-S/ZnS QUANTUM DOTS IN POLYSULFIDE ELECTROLYTE FOR SOLAR CELL APPLICATION

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Solar photovoltaic technology is an important contributor to the reduction of greenhouse gas emissions,[1]and the module efficiency of silicon solar cells is gradually increasing, getting closer and closer to the Shockley-Queisser limit.[2]In addition, efforts are being made to reduce the cost of electricity, and this can be achieved with technological advances in the efficiency of solar cells.[3]To achieve a lower cost per installed capacity, several pathways have been considered, with the objective of achieving a better use of the total energy of thesolar spectrum. In recent years, researchers have been working to optimize each componentof quantum dot-sensitized solar cells (QDSSC). One of the components to be optimized is the electrolyte, which has the function of regenerating the electrons on the surface of the counter electrode and the holes present in the active layer. Therefore, the ideal electrolyte should have the following characteristics: low redox potential, high diffusion coefficient and high electrical conductivity, thus promoting a fast regeneration of the quantum dots and an efficient transport between the electrodes. One strategy to increase electrolyte efficiency is to add additives. In this work, Cu-Zn-In-S/ZnS quantum dots were added to the polysulfide electrolyte in a $CuInS_2$ solar cell. From the results obtained, it was observed that the incorporation of the quantum dots in the electrolyte caused an increase in the efficiency of the solar cell compared to the standard electrolyte.

Acknowledgments:
This work was supported by CNPq, FINEP, CAPES and FAPEMIG.

References:

1. Louwen, A.; Van Sark, W. G. J. H. M.; Faaij, A. P. C.; Schropp, R. E. I. Nat. Commun. 2016, 7, 1.
2. Shockley, W.; Queisser, H. J. J. Appl. Phys. 1961, 32, 510.
3. Moraitis, P.; Schropp, R. E. I.; van Sark, W. G. J. H. M. Opt. Mater. (Amst). 2018, 84, 636.

Influence of solvent vapor and thermal annealing on the morphology and performance of solar cells basedon PTB7-Th:CPDT-4F active layers

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Organic Solar Cells (OSCs) have shown a marked increase in efficiency during the last decade due to the development of non-fullerene electron acceptors. However, for a given choice of donor and acceptor molecules, the optimization of these devices includes post-deposition treatments like solvent vapor annealing and thermal annealing, which can significantly improve the device' s performance. In this work, solar cells with active layers based on PTB7-Th as donor and CPDT-4F as acceptor were produced, and post-deposition treatments as solvent vapor annealing with chloroform (CF) and tetrahydrofuran (THF) and thermal annealing were performed on the active layers. To analyze the impact of these treatments on the morphology, SEM and AFM images were obtained, and both techniques showed significant changes in the morphology when solvent vapor annealing with CF and THF and thermal annealing were applied on the blends of PTB7-Th:CPDT-4F, both separately and consecutively. These post-treatments led to significant changes in the performance of the solar cells with the structure (ITO/PEDOT:PSS/PTB7-Th:CPDT-4F/PFN-Br/Al), with an increase of the Jsc, FF and Voc of the solar cells, which can be directly attributed to the changes in morphology observed by AFM and SEM. These results point to the importance of these post-deposition treatments to further optimize the active layer morphology, and consequently the solar cell efficiency.

Acknowledgements
We gratefully acknowledge financial support from FAPESP (processes 2022/15666-3 and 2014/50869-6) and CNPq (grants 315772/2020-4 and 442239/2017-3). We also thank Dr. Marcelo A. P. da Silva and Manoel R. Roncon for AFM and SEM measurements, respectively.

Isomeric spacers influence on crystal growth and optoelectronic properties in 2D hybrid organic- inorganic perovskites

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Hybrid organic-inorganic perovskite (HOIPs) represents a new class of materials with outstanding physical properties. These 2D materials possess a hydrophobic layer composed of organic spacer cations that offer resistance to heat and moisture. By virtue of their structural and electronic properties, organic spacers are crucial for engineering 2D perovskites, since they can act as a quantum well and also modulate the stability and optoelectronic efficiency. Nonetheless, systematic investigation on the structural and electronic roles is still limited and has not been extensively studied. With that in mind, we have carefully selected two structural isomers n-butylamine (BA) and isobutylamine (iBA) as cation spacers to synthesize 2D halide HOIPs. Such organic spacers are primary amines that satisfy the condition to create van der Waals interface within inorganic layers to achieve the desired 2D perovskite framework. Interestingly, the inductive electronic effect in iBA is an additive to better stabilize a proton, thereby ensuring effective non-covalent interactions through H-bonds. Thus,we have synthesized 2D HOIPs, via solvothermal method, aiming the stoichiometry $BA_2MAPb_2Cl_7$(1) and $iBA_2MAPb_2Cl_7$(2). The XRD patterns exhibit coexistence of Bragg's plane reflections at low angle that suggest contribution of different crystalline phase for (1) and (2). The observation of (200) and (400) planes confirms the growth of an orthorhombic crystal phase associated with the presence of two octahedral sheets building blocks of $[PbCl_6]^{4-}$(n = 2). Extra Bragg's reflections at low angle disclose the presence of a secondary and minority crystal phase (002) without MA molecules. Scanning electron microscopy images analysis show square-like microplates for (1) with the of ~ 20 μm, while rhombus-like planes of 50 μm are observed for (2). The ongoing charge transport and photoluminescent results will be rationalized and discussed considering the influence of organic spacer configuration.

Life Cycle Analysis of Solar Photovoltaic Energy Generation Materials: case study in the floating solar photovoltaic plant at the Santa Clara, Paraná Hydroelectric Powerplant

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In the last decade, the consumption and the installed capacity of photovoltaic generated electricity have reached a high rate of growth. The significant increase in energy conversion efficiency of solar panels and the relative decrease in equipment and system installation costs, both for industrial and residential customers, contributed to this growth. At the same time, it encouraged the search for alternatives to minimize the use of productive areas or areas distant from transmission lines. Consequently, hybrid generation systems were developed taking advantage of hydroelectric powerplant dams, given the advantage of already having the distribution infrastructure in addition to increasing the energy production efficiency of the panel due to the environment water cooling effect. Although considered as a clean form of energy generation, the analysis of its operation must be carried out (from cradle to grave) adopting the concept of cyclical economy, assessing the environmental impact of all stages of the system's useful life. In this investigation, a life cycle analysis (LCA) study was carried out, considering the impact caused by all components of a floating solar photovoltaic powerplant located in the Santa Clara hydroelectric plant dam, located on the Jordão River, between the cities of Candói and Pinhão, in the state of Paraná, Brazil (FloatPV SC), from its manufacturing, assembly, commissioning, use, disposal and/or recycling of components. The objective was to assess the real environmental impact caused, using the SimaPro® software tools, with updated inventory libraries and interfaces, where the segmentation of the plant components was considered, which are: the solar panels, the support structure, the floats, mooring systems, and inverters. In the research, the recommendations in the NBR ISO 14044 (Requirements and Guidelines) and 14040 (Principles and Structure) standards were used.

Mechanisms of Luminescence in RE-doped BaZrO3

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Investigations of BaZrO3 Perovskite compounds suggest that the band-gap and its trappings are largely responsible for luminescence emissions, and their shape can be controlled by the conditions of synthesis of nanocrystals by the Microwave-Assisted Hydrothermal Method (MAH) [1-2]. Crystals doped with rare earths have received considerable attention due to the allowed 5d-4f transition, such as trivalent cerium (Ce3+) with good light yield and energy resolution achieved in many materials, having intrinsic phenomena such as luminescence or fluorescence by conversion energy [3]. This work aims to investigate the properties of BZO Perovskite polycrystals doped with rare earth, synthed using the HAM method, in order to understand their luminescence mechanisms resulting from the energy conversion process. BaZrO3 samples were synthed by the MAH method and characterized using XRD and SEM. XRD results indicated the majority crystalline phase of BaZrO3 with a cubic lattice. SEM images show the present shape and regular from undoped samples. BaZrO3 samples will be synthed and doped with RE, to the purpose of allowing the identification of changes in electronic structures, the influence on the synthesis process, establishing correlations with their optical properties, investigating energy conversion processes compared to pure BaZrO3 and to study potential applications in thin films for solar cells, to be analyzed through of photoluminescence, fluorescence, X-ray absorption and others measurements.

Acknowledgements: CAPES, CNPQ, FAPERGS, UFPEL, CCAF, UFS and PPGFIS.

[1] M. L. Moreira et al. Scripta Materialia 64, 118-121 (2011).

[2] R.U. Fassbender et al. Phys. Chem. Chem. Phys., 17, 11341-11349 (2015).

[3]R.U. Fassbender et al. Journal of Electron Spectroscopy and Related Phenomena 251, 147106 (2021).

OBTAINING P3HT NANOFIBERS WITH PCBM FOR ACTIVE LAYERIN BULK HETEROJUNCTION SOLAR CELLS

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Today, there is a need to increase the photovoltaic parameters (such as efficiency and fill factor) of bulk heterojunction organic solar cells (OPV) and to increase the thickness of the active layer without negatively affecting exciton recombination. One of the alternatives is to place nanofibers of the electron donor polymer and the acceptor material inside the active layer. The objective of this work is to obtain nanofibers containing poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl C61-butyric acid methyl ester (PC61BM) from coaxial electrospinning. These nanofibers will be used as a component of the active layer in BHJ-type solar cells. The polymer used as a shell layer is polycaprolactone (PCL), and the core layer is P3HT with PCBM. The parameters studied in the electrospinning process were the pump''s flow rate used in core-shell-layer solutions and the distance between the needle and collector. The flow rate of the core layer solution had values of 0.1 ml/h, 0.2 ml/h, and 0.3 ml/h. The values of 0.5 ml/h and 1.0 ml/h were employed for the shell layer solution. The applied voltage was 15 KV. The shell layer was removed by immersing the nanofibers in acetone for 24 h. After this, the samples were dried in an oven at 70°C to eliminate all traces of the solvent. The quality of the nanofibers was analyzed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) in the presence and absence of the shell layer. In addition, FTIR spectroscopy of the nanofibers was performedto verify the complete absence of the shell layer.

Acknowledgments:Coordenação de Aperfeiçoamento de Pessoal de Nível Superior, CAPES Foundation

Optical properties over time of cesium-formamidinium lead halide perovskite.

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Solar cells based on lead halide perovskites as an absorber material have shown high efficiency values of around 25%[1]. However, a major disadvantage of this material is its degradation over time, which considerably reduces the useful life of devices. One way to overcome this problem is by inserting organic cations and inorganic ions to increase the stability of the material [2]. In this work, we deposited the hybrid perovskite FA0.83Cs0.17Pb(Br0.17I0.83)3 and investigated its stability through optical measurements over time. One advantage of using optical characterizations for material investigation is the possibility of obtaining interface information without damaging the sample [3].

The perovskite was deposited on FTO (TEC7) using chemical route and spin-coating. Transmittance and reflectance measurements (specular and diffuse) were made for light falling on both sides of the samples. For the pristine deposited samples the maximum light scattering were 8%, which occurs preferentially in the perovskite grain boundaries. The Urbach energy obtained was in the order of 17-18 meV, indicating low disorder. Measurements over time showed a 35% drop in absorbance after 21 days, while the classic perovskite CH3NH3PbI3 (MAPbI3) deposited for comparison reached similar values in the first 72 hours, indicating greater stability of perovskite FA0.83Cs0.17Pb(Br0.17I0.83)3. The bandgap remained practically constant at 1.65 eV. The Urbach energy increased, reaching a value of 20-21 meV, associated with an increase in the disorder at the grain boundary, indicating a preferential degradation in this region.

Acknowledgements:
The authors thank CAPES (88887.817534/2023-00) and FAPESP (2020/12356-8) for financial support.

References:
1. Laboratory National Renewable Energy, Https://Www.Nrel.Gov/Pv/Assets/Pdfs/Best-Research-Cell-Efficiencies.20200104.Pdf 1 (2020).
2. M. Saliba and T. Matsui, Energy Environ Sci 9, 1989 (2016).
3. B. Zhao and M. Abdi-Jalebi, Advanced Materials 29, (2017).

ORGANIC SOLAR CELLS: STUDY OF STABILITY AND DEGRADATION UNDER CONTINUOUS USE

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Organic solar cells (OSCs) are devices that convert light energy into electric current using small molecules or conjugated polymers [1]. Despite their high efficiencies, there is a lack of studies on the degradation of OSCs under continuous use and ambient conditions. In 2011, testing guidelines and protocols were established during the International Summer on Organic Photovoltaic Stability (ISOS) to standardize OSC reliability/stability tests [2]. However, many in the OSC community still do not follow these protocols, making it difficult to compare and replicate degradation data. This work focuses on studying degradation phenomena in OSCs under continuous operation following ISOS protocols, using a P3HT:PC61BM polymer blend in a conventional BHJ structure, J-V measurements were obtained under illumination, and degradation was measured using two ISOS protocols: ISOS-D1 and ISOS-L1. Results showed that the device retained 80% of its power conversion efficiency (PCE) after 3.5 hours (T80) for ISOS-D and after 0.5 hours (T80) for ISOS-L, with oxygen identified as a key factor in degradation. The guidelines for improving the lifetime of OSCs under continuous operation are discussed.

Acknowledgements: CAPES, CNPq and National Institute of Organic Electronics (INEO)

References:
[1] Doumon, N. Y. (2019). The degradation of organic solar cells: from chemistry to device physics through materials.
[2] Reese, M. O. et al (2011). Consensus stability

Perovskite semiconductor cross-linking polymernetwork passivation for tandem photovoltaics

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Wide bandgap lead-halide perovskite (LHP) materials appear as ideal semiconductor for tandem architectures with silicon bottom cells due to their large absorption coefficient and processing compatibility. However, LHP still suffer from strong degradation issues mostly due film inhomogeneities, pinholes and surface and electronic grain boundary defects. The most successful strategies to passivate crystalline defects in LHP include non-stoichiometric passivatio filling halide vacancies by anio physical passivation, dimensional engineering with 2D or 1D heterojunctions and passivation with cross-linking agents. Herein, we demonstrate an LHP passivation strategy based on the formation of an ionic polymeric network obtained by thermal-induced. In the developed process, an ionic monomer is deposited on the surface of perovskite surface after thermal annealing. This precursor upon heating undergoes a polymerization reaction forming a cross-linking polymeric network that, simultaneously, passives defects at the grain boundaries and on the surface of the perovskite film. Using a perovskite composition Cs0.85DMA0.15Pb0.9Sn0.1I3 (Eg = 1.64 eV) optimized for tandem applications three different styryl terminated monomers were evaluated. All monomers presented strong perovskite passivation effect enormously increasing the environmental stability without altering the semiconductor bandgap, morphology and absorption spectrum.

This work was supported by the grants #406558/2022-1 (CNPq) and #442553/2019-6 (SisNano/CNPq) and BYD Energy do Brasil (PADIS-Program).

[1] Yusoff, A. R. bin M. Vasilopoulou, M. Dimitra, Georgiadou, D. G. et. al. Energy Environ. Sci., 14, 2906-2953, 2021.
[2] Li, C., Li, H., Zhu, Z. et. al. Sol. RRL, 5, 2000579, 2021.

Phase-pure Ruddlesden-Popper's Perovskites Electroluminescent Devices

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2D organic-inorganic hybrid perovskite structures are attractive soft crystalline materials to be used in optoelectronic devices, such as photovoltaics (PVs) and light-emitting diodes (LEDs), mainly due to their capability to improve devices stability against open environmental conditions (humidity and oxidative atmosphere) and the formation of quantum well (QW) electronic structures [1]. In particular, lead-halide Ruddlesden-Popper phases (RPs) are great candidates to achieve such optoelectronic features and, at the same time, protect them from humidity, increasing perovskite stability. Herein, we propose a low-cost and straightforward methodology to process butylammonium-based 2D lead-halide perovskite materials for efficient LEDs active layers [2]. The BA_2PbBr_4(blue), and $BA_2[FAPbI_3]PbI_4$(red) RPs were solubilized into acetonitrile and spin-coated as emissive layers (EML) in an ITO|PEDOT:PSS|PVK|2D-RPs|TBPi|LiF|Al LED structure. Time-resolved and steady-state photoluminescence experiments, combined with atomic force microscopy and structural studies of the RPs thin-films over poly(N-vinylcarbazole) PVK, revealed important insights into the morphology, charge-carrier dynamics, energy, and charge transfer from PVK polymer to the RPs layer The LEDs exhibited a roll-off with brightness and EQE values around 1300 cd $m^{-2}$and 4 %, respectively. This new synthetic route, combined with simple solution-processable tools, allows obtaining high-color purity, low-cost, and functional 2D-perovskite LEDs opening further frameworks for using such kinds of perovskite materials as new active matrixes for optoelectronic devices

References

[1] M. S. Holanda, R. F. Moral, P. E. Marchezi, F. C. Marques, A. F. Nogueira, EcoMat 2021, 3, 1
[2] R. F. Moral, L. G. Bonato, J. C. Germino, W. X. Coelho Oliveira, R. Kamat, J. Xu, C. J. Tassone, S. D. Stranks, M. F. Toney, A. F. Nogueira, Chem. Mater. 2019, 31, 9472.

Photocurrent Effects in Ultrasmall Quantum dots CdSe/CdS

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Ultrasmall quantum dots (USQDs) are rarely reported in the literature, they have s smaller than 5 nm, excellent optical properties and good biocompatibility. A not analyzed effect in USQDs is photocurrent. Photocurrent occurs when photons are exposed with energy large than the band gap, thus producing charge carriers. With a built-in field or an externally applied bias, these charge carriers can be separated, thus producing a photocurrent in an external circuit. Here we use two types of USQDs, one CdSe (1ST) and another CdSe/CdS core/shell (9S), to analyze photocurrent effects not seen before in USQDs. We used the Raman spectrum to confirm the presence or absence of the shell. The photocurrent effect in USQDs 1ST showed the pattern photocurrent effect for semiconductor materials. Turning on the light the sample had a rise time of 1.26 s and when the light was turned off, it had a decay time of 0.70 s, with the maximum current value being 12.90 pA. However, the 9S sample showed an overshoot when the light is turned off. This effect is related to the accumulation of charge carriers that appear when QDs are subjected to an electric field and the presence of light, this accumulation caused by the potential barrier formed by the shell is released when the light is turned off. When the light is turned on, its rise time is 2.28 s, when the light is turned off, the decay time is 2.59 s, with a maximum current value of 70.51 pA.

Pressure-induced Changes in Optical and Structural Properties of 2D and 3D Halide Perovskites for Energy Conversion Technologies

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Hybrid organic-inorganic halide perovskites and their low 2D dimensional counterpart have attracted great attention due to their outstanding optoelectronic properties. These compounds can be used for light emitting and solar cell devices. Coupling between electrical and optical properties leading to high conversion efficiency is close related to crystal distortion. Controlled manipulation of the crystal distortion and compression by external pressure can be a rational route to understanding the relationship among optical, charge transport, and structural properties. Here, we have synthed Ruddlesden-Popper samples ($BA_2(CH_3NH_3)_{n-1}Pb_nI_{3n+1}$) by using N-butylamine (BA) molecules as a spacer for *n*= 1, 2, and 7 (close to a 3D system) and its tridimensional counterpart ($MAPbI_3$, MA= CH_3NH_3) to study the optical and structural evolution under pressure up to ~12 GPa. Optical images showed microplates morphology for 2D samples, unlike 3D, which exhibited cuboid morphology. The photoluminescence of 2D crystals displayed a unique behavior that can be characterized by three distinct regimes when pressure is applied. Initially, these crystals presented a single excitonic peak that is characteristic of its respective n value. However, at intermediate pressures ranging from 1.5 to 8 GPa, the PL peak is splitted into higher photon energy. In the high-pressure regime, the PL emission is completely quenched. Notably, only *n*=7 crystals exhibited complete reversibility after decompression. In contrast, the 3D perovskite exhibits a rapid decline in emission marked by PL quenching when subjected to 3 GPa. Rietveld refinement analysis of the data under pressure are going on and will be correlated to the induced and quenched PL emitting peaks. This study intends to reveal whether the quasi-3D (*n*=7) sample is chemically more stable than MAPbI3 and preserving the charge transport efficiency, which may represent an alternative for use in energy conversion devices.

Slot Die Coating of SnO2 Selective Contacts forPerovskite Solar Cells.

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Perovskite solar cells (PSC) are in the spotlight among the energy conversion technologies. Nowadays, n-i-p devices surpass 25% of efficiency and tin oxide (SnO_2) is commonly used as a selective electron transport layers (ETL) [1]. For commercialization, the improvement of stability and the scalability of the devices are current challenges. For commercial production, the deposition of all functional layers needs to be upscalable and, ideally, low-cost and material-efficient. Among the processes, slot-die-coating [2] is very attractive as it allows coating with high-throughput many kinds of materials (including the perovskite layer) using a solution as a precursor and adjusting a few parameters, such as the concentration of the solution, the substrate speed, and the solution pump rate.

In this work, the deposition of SnO_2 by slot-die coating is presented aiming for the application as ETL layer in perovskite solar cells. As a starting point, the same precursor solution as optimized for spin-coated layers were used [3]. The substrate dislocation speed and slot-die head-substrate distance were constant, and the pump rate was changed, aiming to achieve a 30 nm uniform coating of SnO_2. SEM measurements were used to analyze the thickness and morphology. For solar cells devices based on the slot-die coated SnO_2 layers in combination with a FACs-based perovskite absorber were used. J-V scans were performed to evaluate the efficiency and MPP tracking was carried out to test device stability.

Acknowledgements:CAPES - PROEX Scholarship, CAPES-PrInt Process No: 88887.571101/2020-00. FAPESP Process No: 2020/12356-8. Ms Carola Klimm for the SEM measurements.

References

[1] Min, H. et al. Nature, 598 (78881). 2021.
[2] Whitaker, J.B. et al. Sustainable Energy Fuels 2. 2 (11). 2018
[3] Dagar J. et al. Solar RRL, 3 (9). 2019.

Stability of metal contacts to typical processing conditions of perovskite solar cells

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Charge collection in large-area perovskite solar cells is achieved by using a metallic contact in at least one side of the cell (top or bottom contact), and Ag is often the metal of choice. However, this material suffers from progressive degradation due to reaction with iodide species from the perovskite layer. Thus, the search for a substitute metal that could show adequate cost, conductivity and chemical stability is imperative for the industrialization this technology. Herein, metal grids were deposited by sputtering onto 5x5 cm^2FTO-glass substrates, aiming at their use as front contact in large-are single cells. Different metals were investigated, which were selected based on their current applications in the photovoltaic or electronic industries: Ag, Al, Au, Cu, Mo, Ni, Pd, Pt, Ta. As-deposited grids exhibited good adherence to the substrate, and the lines were continuous, without pinholes, as observed by scanning electron microscopy (SEM) and optical microscopy (OM). Then, the grids were subjected to different steps of a typical perovskite cell assembly procedure: a) cleaning step; b) UV-ozone treatment; c) heating at 180 °C for 1 hour (mimicking the treatments applied for SnO_2ETL deposition); and d) heating at 100 °C for 1h (mimicking the perovskite annealing step). SEM, OM and resistivity data were collected after each step, and after performing a sequence comprising of all 4 steps, which would be used in a complete cell assembly. Afterwards, the stability of the metals when in contact with the SnO_2layer or with direct contact with the perovskite layer, and after accelerated aging tests, was also assessed. The data gathered reveal that most of the metals suffer some sort of degradation in at least one of the conditions investigated, showing that a careful selection of the metal contact is of utmost importance for long-term use of these cells.

Acknowledgments: PCI/CNPq (301138/2021-4), FAPESP (2017/11986-5) and Shell.

Study of the degradation mechanisms of the CuS counter electrode in CdS quantum dot sensitized solarcell

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The need for renewable energy sources, with less environmental impact and low cost, has been extensively studied within Materials Science. Alternatives that prove to be effective and capable of large-scale application are sought, with quantum dot-sensitized solar cells (QDSSCs) being a viable alternative [1]. However, for these cells to leave the academic environment and be applied in everyday life, it is necessary to improve stability. Given this context, the focus of this study was to investigate the stability of the CuS counter electrode (CE) in CSSPQs [2]. The CuS CE were deposited by the chemical bath deposition technique. Synthesis and deposition parameters were investigated: reagents concentration, synthesis temperature and post-synthesis treatment. QDSSCs were prepared using CdS quantum dots and characterized through IxV curves and impedance spectroscopy, for 43 days. Degradation mechanisms that reduce the performance of QDSSCs and CuS CE was investigated. Adequate adjustment of the CuS synthesis and deposition parameters were extremely relevant to improve devices stability. Stable devices, for 43 days, was obtained, for CuS deposition in the optimal conditions. The aging study showed that there was a corrosion of CE in the electrolyte and possible diffusion to the photoanode, causing a reduction in the CE electrocatalytic activity and consequently an increase in the CE charge transfer resistance. It was concluded that one of the relevant factors in the QDSSC performance degradation is related to CE charge transfer resistance, due to the CE instabilities. To increase the overall stability of QDSSCs, it is vital to understand the degradation mechanisms in order to prove their reliability.

Acknowledgments:

We thank UFSJ for the project opportunity. This work was supported by CAPES, CNPq e FAPEMIG.

References:

[1] A. S. Rasal, S. Yadav, et al., Nano Energy, 94, 106854 (2022)

[2] A. S. Rasal, M. Wu, et al., Chemical Engineering Journal, 446, 137293 (2022)

STUDY OF THE STRUCTURAL STABILITY OF MAPI ON THE MAPI/P3HT INTERFACE DEPOSITED BY SPIN-COATING

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In this work, the structural and photophysical stability of MAPI was analyzed (at the MAPI/P3HT interface. Water is the main catalyst for the irreversible degradation of perovskite material, pointing to a problem for the lifetime of these solar cells. The deposition of Poly(3-hexylthiophene) P3HT on the perovskite had the main purpose of providing a form of encapsulation for the MAPI, that is, to prevent the device from being affected by the humidity of the environment causing degradation. The MAPI was deposited on the FTO substrate (fluorine-doped tin oxide) in two rotation steps. Chlorobenzene was used as an antisolvent and after deposition, the sample was heated at a temperature of 100°C until the transition to the crystalline phase occurred. To form the MAPI/P3HT interface, a solution of P3HT diluted in chlorobenzene was deposited on the MAPI by spin-coating. Photoluminescence (PL), UV-vis absorbance, and x-ray diffraction (XRD) measurements were taken over 30 days. Using the x-ray diffraction technique (XRD), we followed the degradation process of the encapsulated sample and the same one without encapsulation, exposed to the same environment, allowing us to analyze the variation in the intensity of the peaks referring to the product of the decomposition reaction, PbI2. The DRX data showed that over the course of 1 month, there was better stability in the MAPI/P3HT interface. The photoluminescence measurements indicate that the excitation and emission bands of the P3HT polymer are different from the characteristic of MAPI, thus we do not have overlapping bands that could decrease photon absorption. Finally, the MAPI/P3HT interface proved to be efficient in terms of stability for future applications in photovoltaic devices.

Acknowledgments: CAPES, FAPEMAT, and CNPq.

Study Of Type I Architecture in MAPbI3 based solarcells with MoS2 Quantum Dots

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A sustainable approach to meet the global energy needs require research efforts to improve our energy conversion technologies and reliance on renewable and clean sources. Organic-inorganic halide perovskites solar cells (PSCs) have received great interest in solar energy conversion due to their high efficiency, low exciton binding energies and high light absorption [1]. However, hysteresis phenomena, chemical stability of perovskites exposed to ambient conditions, make their long-term use challenging. Low dimensional materials have attracted a lot of interest due to their fascinating properties and unique structure [1]. As a way to explore the use of these materials and their effects on the properties of the PSCs, MoS2 quantum dots (QDs) have been integrated in the absorber layer of PSCs. The aim of this work was to study perovskite-MoS2 QDs composite films and their atypical alignment of energy levels, forming a type-I heterojunction, which has not yet been extensively explored [2]. A type-I architecture makes it possible to enhance carrier extraction from the QDs and, in general, to exploit synergies between the respective material qualities. A detailed characterization of the composite film materials and film properties through test structures of photovoltaic devices will then be presented. The electronic band structure of the MoS2 QDs and MAPbI3 films has been experimentally determined, using a combination of techniques that revealed the type-I alignment.

Acknowledgment: This work has been supported by CAPES and CNPq.

References:

[1] Luo, Z. et al. Enhancing the Efficiency of Perovskite Solar Cells through Interface Engineering with MoS2 Quantum Dots. Nanomaterials, v. 12(17), p. 3079 (2022).

[2] Rocks, C. et al. Type-I alignment in MAPbI3 based solar devices with doped-silicon nanocrystals. Nano Energy, v. 50, p. 245 - 255 (2018).

Synthesis and Characterization of Ternary QuantumDots Obtained via Hydrothermal Route Applied in Sensitized Solar Cell

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Abstract: With the rapid development of the world economy and the increase in population, increased energy demand and environmental pollution, which have become challenging problems for sustainable development. In this context, an attractive alternative that has been developed by researchers to produce renewable energy is the quantum dots-sensitized solar cells (QDSSC), which have a theoretical efficiency of ~44% [1]. Quantum Dots (QDs) are colloidal nanocrystalline semiconductors with a diameter ranging from 2 to 15 nm, whose most prominent property is the quantum confinement effect. The synthesis commonly used to produce these QDs occurs in an organic medium. In this work, QDs Cu-In-S_2(CIS) were synthesized via hydrothermal route, considered an economic and ecological alternative in relation to organic syntheses [2]. The choice of CIS QDs was due to their broad visible absorption spectrum (with an appropriate bandgap of ~1.5 eV) and absence of toxic metal such as Cd or Pb. The QDs were applied in the sensitization of photoanodes for solar cells, which efficiency achieved was of 1.37% with a fill factor of 0.46. Based on the results obtained, the hydrothermal synthesis proved to be a simple and green route for the obtention of high-quality nanocrystals for photovoltaic applications. Other synthetic parameters of the CIS QDs will be discussed relating to the efficiency of the solar cells achieved.

Acknowledgments

This work was supported by CNPq, FINEP, CAPES and FAPEMIG.

References

[1] Sahu, A., Garg, A., Dixit, A. A review on quantum dot sensitized solar cells: Past, present and future towards carrier multiplication with a possibility for higher efficiency. Solar Energy, 203, 210-239, 2020.

[2] Jain, S., Bharti, S., Bhullar, G. K., Tripathi, S. K. Synthesis, characterization and stability study of aqueous MPA capped CuInS2/ZnS core/shell nanoparticles. Journal of Luminescence, 252, 119279, 2022.

Synthesis and physical properties of 2D BA2PbX4 nanoplatelets and bulk halide perovskites

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Lead halide perovskites have been attracted great attention as light emitting materials due to the unique optoelectronic properties such as high photoluminescence quantum yield (PLQY) and long PL lifetime. The fabrication of UV-light emitting device by using this perovskite family with high efficiency can be used in interdisciplinary fields. Nowadays, the biggest challenge is to synthe these materials emitting in the UV region through anion exchange, finite effect, and/or changing dimensionality. The 2D perovskites have recently emerged as a family of more stable materials that present peak emission in the UV region. In this work, 2D halide perovskites of (M)2PbX4 were studied, where M are molecules acting as a spacer between the perovskite layers formed by the confined [PbCl6]4- octahedrons. The inorganic layer shows strong confinement effect enlarging the band gap energy and improving radiative exciton recombination. Here, the strategy used was anion exchange by using I, Br, and Cl in (BA)2PbX4 and using N-butylamine (BA) molecules as spacers. Structural and morphological characterizations indicated that all the 2D perovskites samples were successfully synthed resulting in quantum nanoplatelets. The observed Bragg reflections can be indexed as belonging to an orthorhombic crystal structure with a Pbca space group symmetry. Scanning and transmission electron microscopy show a laminar 2D structure indicating the formation of nanoplateles which have two distributions 9 nm and 36 nm. Photoluminescence measurements reveal emitted photon energy around 3.57 eV for nanoplatelets and E1 = 2.38 eV, E2 = 2.88 eV, and E3 = 3.47 eV for X = I, Br, and Cl, respectively. The (BA)2PbCl4 bulk sample showed a very low PL intensity while quantum nanoplatelets, combining and structural confinements revealed improved UV emission with higher PL quantum yield. We will present a systematic study involving synthesis and physical properties of these 2D UV emitting materials.

Synthesis and Physical Properties Study of 2D Hybrid Perovskites for Energy Conversion

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Organic-inorganic halide perovskites and their low 2D dimensional counterpart have attracted a lot of attention in the development of new optoelectronic technologies. The two-dimensional Ruddlesden-Popper (RP) perovskites have crystal structure versatility and improved long-term stability when subjected to light, moisture, and thermal stress. These perovskite crystals are structured as n-layer inorganic octahedral sheets, $(OM)_2(CH_3NH_3)_{n-1}Pb_nI_{3n+1}$, sandwiched by two layers of large organic molecules (OM) spacer acting as quantum well barriers. Here, we have synthed RP samples by using N-butylamine (BA) molecules as a spacer $BA_2MA_{n-1}Pb_nI_{3n+1}$ for n = 1, 2, 3, 4 and n = ∞ (3D) and $MA=CH_3NH_3^+$(methylammonium). Powder X-ray diffraction indicated that the samples for n = 1, 2, 3 and 4 crystallizes in an orthorhombic crystalline structure while n = ∞ shows Bragg reflections belonging to a tetragonal structure. Scanning electron microscopy images confirm 2D morphology. They present a laminar structure with the thickness of the samples increasing with the value of n from 5 μm (n=1) to 15.8 μm (n=4). The 3D structures have irregular rounded particles, with an approximate between 25 μm to 60 μm. The quantum confinement effect presented in the octahedral sheets leads to a blue-shifted emission peak. Thus, we have observed an emitted photon energy progressively decreasing from 2.38 eV (n=1) to 1.50 eV (n=∞). Electrical conductivity measurements, with light and without light, as a function the number octahedral sheets are being carried out in order to observe the effect of quantum confinement on the photoinduced charge carriers. The study of the structural, morphological, optical, and electrical properties of these 2D samples will berationalized involving separation and charge transfer processes. Our study may reveal if these 2D systems can represent a possible alternative for use in energy conversion technologies.

Synthesis of High Quality Zn-Cu-In-S/ZnS Quantum Dots and the Ligand Exchange Effects on SensitizedSolar Cell

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Solar cells have gained increasing interest in recent years, as they represent the most important way of converting sunlight into electricity. Quantum dots sensitized solar cells (QDSSC) have attracted attention due to their low manufacturing cost and high solar energy conversion efficiency [1]. Quantum dots (QDs) are colloidal nanocrystalline semiconductors, the Zn-Cu-In-S (ZCIS) and Zn-Cu-In-S/ZnS (ZCIS/ZnS) QDs stand out for presenting advantages due to high optical absorption coefficients, high quantum yields, greater stability, decrease in surface defects and low toxicity [2]. In order to control the hydrodynamic particle , it is necessary to exchange the surface ligands. In organic syntheses, the most used ligand is dodecanethiol (DDT), whereas to disperse nanoparticles in an aqueous medium, the most used ligands are thioglycolic acid (TGA) and mercaptopropionic acid (MPA). In this work, QDs ZCIS and ZCIS/ZnS were synthed in organic medium and both QDs had their surface modified by exchanging MPA and TGA ligands, in order to achieve stability in aqueous medium. The best result obtained was the cell sensitized with QD CZIS-TGA, obtaining 2.9% efficiency. Based on the results, it was possible to perceive that the ZnS shell hinders the transfer of charge. In relation to the surface ligands, the TGA proved to be more efficient than the MPA, indicating a greater amount of adsorbed QDs due to its reduced .

Acknowledgments

This work was supported by CNPq, FINEP, CAPES and FAPEMIG.

References

[1] Song, H. et al. Zn-Cu-In-S-Se Quinary “Green” Alloyed Quantum-Dot-Sensitized Solar Cells with a Certified Efficiency of 14,4%. Angewandted Chemie, 60, 11, 6137-6144, 2021.

[2] Zheng, Y., Sadeghimakki, B., Brunning, A. L. J., Piano, M. E., Sivoththaman, S. Emission and decay lifetime tunability in less toxic quaternary ZnCuInS quantum dots. IEEE Trans Nanotechnol, 20, 525-533, 2021.

Temperature dependence of the photoluminescence and exciton-phonon coupling in low-dimensional metalhalide perovskites

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Low-dimensional metal halide perovskites (LDMHPs) have captured considerable attention due to their promising optoelectronic properties. The excitons in these materials are strongly confined, resulting in large binding energies due to the significant quantum confinement and reduced dielectric screening. Excitons in LDMHPs can exist in two forms: free exciton polaron (large polaron) or self-trapped exciton (STE) (small polaron). Free excitons display narrow, near-band edge emissions, while STEs are characterized by broad emissions that are tightly localized by lattice distortion.

This study focuses on investigating the photoluminescence (PL) spectra and the exciton-phonon coupling in LDMHPs at varying temperatures. The analysis of the temperature-dependent PL spectra reveals that STEs dominate the low-temperature PL emission, while band-to-band excitons become more prominent at higher temperatures. Additionally, the coupling strength between STEs and phonons is investigated as a function of temperature, leading to a shift in the STE emission.

Our findings provide insight into the coexistence and complex interplay between long- and short-range interactions in LDMHPs, which present significant challenges in understanding and describing exciton behaviors in these materials.

The dark I-V behavior of a PCE > 20% perovskite solarcell and a Nb_2O_5/PEDOT Schottky diode modified by a PMMA dopped $Ti_3C_2T_x$-MXene passivation layer

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Perovskite solar cell (PSCs) has become a promising photovoltaic technology. Despite achieving a conversion efficiency exceeding 25.5%, reaching 20% remains a significant challenge [1], which reveals the still incomplete knowledge regarding the understanding of performance-limiting mechanisms [2]. Here, we propose a method based on the dark current-voltage (I-V) behavior to evaluate the effect of a PMMA-MXene passivation layer on the PSCs. We also applied it to a simpler Nb_2O_5/PEDOT Schottky diode (SD). The passivation layer was deposited via spin coating between the perovskite film and the ETL in a p-i-n architecture and between the Nb_2O_5film and the PEDOT top electrode in a sandwiched diode structure. We fabricated the PSC following a meticulous protocol under glove box conditions, whereas the Schottky diode was partially fabricated under room conditions. For SDs and PSCs, the passivation layer prevents a portion of the current shunt with a minor drawback in series resistance. We extracted the ideality factor (IF) using analytical methods, in which the SDs exhibited an increase after the addition of the passivation layer (3.2) compared to the control device (2.7). For the PSCs, an initial IF increase after adding the PMMA layer (from 2.7 to 3.7) was followed by a decrease upon the incorporation of the MXene (3.0). This trend was confirmed by the light intensity-dependent open-circuit potential (Voc) analysis, i.e., an IF value shift from 1.9 to 3.0 followed by a decrease to 1.85 after MXene. Therefore, the passivation maintains the benefits of the PMMA layer while providing additional advantages in electron collection due to the MXene. The dark I-V and IF analysis provide insights that may offer a fresh perspective on the analysis and optimization of PSCs.

Acknowledgements:

FAPESP (22/10998-8 and 20/12356-8), CNPq, Capes, FINEP (01.22.0289.00 (0034/21)).

References:

[1] T. Wu and L. Han, 13, 152 (2021)

[2] D. Glowienka and Y. Galagan. 34, 2105920 (2022)

The Electron Pair Distribution Function (ePDF) for Structural Studies of Cadmium Selenide Quantum Dots

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Quantum Dots (QDs) like Cadmium Selenide (CdSe) are nanometric semiconductor materials under massive study due to their optical and electronic properties controlled by quantum confinement[1]. Nanoparticle crystallographic structure can hinder the efficiency of QDs applications, mostly due to the concentration of defects such as grain boundaries, stacking faults, and vacancies. The small size nanoparticle of QDs makes it more difficult to characterize the crystallographic structure by standard techniques like X-ray Diffraction. Then, the Pair Distribution Function (PDF) analysis of the total scattering data has beenraised as an alternative method to suppress those problems and solve the atomic arrangement structure for nanomaterials[2]. Alternatively, the PDF from electron diffraction (ePDF) presents itself as an optimal possibility to characterize nanoparticles as the strong electron matter interaction enables the acquiring of diffraction patterns for a known ensemble of nanoparticles, i.e. TEM equipment can collect diffraction by a precise nanoparticle region matching images and elemental composition analysis with ePDF analysis. In this work, CdSe QDs from 1.9 nm to 4.0 nm were synthed by modifying the procedure. A modified code of eRDF Analyzer software was used to calculate the structure factor S(Q) by normalizing the intensity scattering profile I(Q) with the expected atomic scattering factors. The Reduced ePDF G(r) was obtained by the Fourier Transform of S(Q). Simulated I(Q), S(Q), and G(r) for spherical z-CdSe nanoparticles were introduced in the eRDF code to guide the data treatment and extraction of the final G(r). We have seen that G(r) results lead to smaller crystallite compared to TEM morphological diameter and the obtained from the position of the first excitonic peak. Concluding, ePDF G(r) showed to be a good crystallographic tool for semiconductor CdSe QDs analysis.

[1] García de Arquer *et al., Science* **373**, 640, 2021
[2] *Matter*4, 441-460, 3, 2021

Thermal Stability of Thermally Evaporated BiI_3 ThinFilms

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The current high demand for cheap and renewable energy has given a great deal of support for the development of low cost photovoltaic solar cells. Today the most promising material is the lead-halide perovskite that has achieved efficiencies as high as 25.8% in few years of research. In spite of the incredible efficiency obtained, it faces a problem in stability, apart from having lead, a toxic element. In this way, some lead-free materials have been studied in the last few years, including bismuth triiodide (BiI_3) [1], a semiconductor with a bandgap of 1,67 eV [2], thereby suitable for photovoltaic applications. Here we investigate the thermal stability of thermally evaporated BiI_3thin films annealead up to 150 °C in ambient atmosphere and in the absence of illumination. BiI_3films show similar X-ray diffratrograms with indexed peaks related to an R-3 rhombohedral crystal structure. Tauc plots of transmittance data of BiI_3in UV-vis range indicates a bandgap of 1.72 eV regardless the annealing temperature. BiI_3morphological properties, observed through SEM images, are also not changed regardless the annealing processes. Thereby, the structural, morphological and optical properties of annealing on thermally evaporated $BiI_3$are not considerably altered, the films retaining their properties when heated up to 150 °C. Since this temperature is higher than the operating temperature of solar cells, these results corroborate for the possible photovoltaic application of BiI_3.

Acknowledgements:

CNPq, Capes, Fapesp, Lamult Unicamp, BYD Energy. References:

[1] R. E. Brandt et al. J. Phys. Chem. Lett., 6, 4297-4302 (2015).

[2] N. J. Podraza et al. J. Appl. Phys., 114, 033110 (2013).

Ti_3C_2Tx-MXene dopped PMMA passivation layer for highly efficient and stable inverted perovskite solar cell

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Perovskite solar cells (PSCs) are currently one of the most promising technologies for generating clean energy. Despite significant advances in conversion efficiency, PSCs face drawbacks regarding their long-term stability and interfacial defects between perovskite and the associated charge transport layers. Recently, PMMA has been used as passivation layer to bypass PSCs challenges, however, its high resistance has compromised the polymer application[1]. MXene, a new class of 2D material, has shown high conductivity, transmittance and has been successfully applied to PSCs in the last years[2]. In this way, a PMMA passivation layer dopped with Ti_3C_2Tx-MXene was deposited via spin coating between the perovskite and electron transport layer of an inverted PSC. The incorporationof MXene was confirmed by XDR patterns and UV-Vis spectroscopy. FE-SEM images show auniform formation of the passivation layer onto perovskite for all samples. The presence ofthe MXene in the PMMA layer resulted in the increase of PCE (21.9%) when compared topure PMMA (17.6%) based devices. Photo-CELIV measurements showed a substantial increase of charge mobility and density of extracted charges after incorporation of MXenes.The presence of the 2D material also resulted in decrease of the series resistance whereasnegligible difference in PMMA shunt resistance was observed. TPV measurements showedenhanced charge lifetime for the MXene based devices. These results were corroborated bythe decrease of the calculated trap density indicating a defect passivation by PMMA:MXenelayer. Finally, PMMA:MXene based device showed substantial improvement of stability bywithstanding 95% of its original PCE against only 87.5% of the pristine device after 3000hours.

Acknowledgements:
To FAPESP (Grants: 2019/2256-7 and 20/12356-8), CNPq, Capes, FINEP (Grant: 01.22.0289.00 (0034/21)) for the financial support.

References:
[1] J. Peng and Y. Wu, 10., 1792-1800 (2017)
[2] L. Yang and P. Li, 81., 443-461 (2023)

L-Radiation Detectors

Crystallographic characterization of the mineral zircon and the thermochronology

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The zircon mineral was characterized by SEM and micro-Raman before and after etching to study its crystallographic structure. Through the etching used to reveal the fission tracks is possible to observe crystallographic damage in the zircon grain that may have been acquired during its genesis or throughout its geological history. These imperfections are known in the literature as Metamictization, Leaching and Chemical Differentiation. The zircon grains were classified, after etching, into four groups: Homogeneou Heterogeneous, Hybrid and Anomalous [1]. Based on this classification, new characterization studies of this mineral were carried out showing nine groups. These results were used to obtain zircon grain age data using two methods simultaneous: the Fission Track Method and the U-Pb method. These ages can help to better understand the thermochronology of the geological region where the zircon grains were collected.

References:

[1] Rosana Silveira Resende, Carlos Alberto Tello Sáenz, Luiz Augusto Stuani Pereira, Elton Luiz Dantas. The effect of Chemical and physical imperfections in zircon grains in influencing U-Pb age analyses: Insights from zircon fission track etching. 2019. Lithos 346-347 105138.

Unraveling the Arrhenius Activation Energy for Fission-Track Annealing in minerals

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Fission-Track Thermochronology (FTT) is a technique used to estimate the thermal histories of minerals and rocks containing uranium, such as apatite, zircon, and epidote. The spontaneous fission of uranium produces two fragments that damage the mineral crystal lattice along their trajectory. At moderate temperatures, these damaged regions, known as fission tracks, are partially restored through annealing. To understand annealing kinetics, mineral samples are heated at varying temperatures and durations. Empirical Arrhenius-type equations are used to relate reduced fission-track length with temperature and duration of heating. These models are extrapolated to the million-year timescale for geological applications [1]. However, current annealing models are purely statistical and do not provide information about specific annealing mechanisms. In this contribution, we present a physicochemical framework that allows us to retrieve the Arrhenius activation energy for annealing mechanisms and derive new models from different combinations of activation energies [2]. This framework has the potential to provide insights into annealing mechanisms and increase the reliability of model extrapolations.

Acknowledgements: This work has been funded by CNPq grant # 305620/2022-3.
Thanks to FAPESP and CNPq for funding this research, and to all who contributed to this work.

References:
[1] Guedes, S., Jonckheere, R., Moreira, P.A.F.P., Hiescher, R., 2008. On the calibration of fission-track annealing models. Chem. Geol. 248, 1-13. chemgeo.2007.10.005.
[2] Rufino, Matheus, and Sandro Guedes. Arrhenius activation energy and transitivity in fission-track annealing equations. Chemical Geology 595 (2022): 120779.

Analysis of phototransferred thermoluminescence inrose quartz

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Due to its luminescent properties, natural quartz is widely studied by stimulated luminescence techniques including thermoluminescence (TL) [1]. TL is a temperature-resolved emission observed from previously irradiated insulators [2]. When some TL glow peaks are removed by heating, it is possible to regenerate them by exposing the sample to light of a specific wavelength for a certain time. This phenomenon is named phototransferred thermoluminescence (PTTL) and occurs because of the transfer of electrons from a deep electron trap to a shallower electron trap, using light [1]. For this study, a powdered rose quartz sample (>75 μm), with mass of 110 mg, was used. PTTL measurements were carried out using a Risø TL/OSL DA-20 luminescence reader. Sample were irradiated to 130 mGy at room temperature using a built-in 90Sr/90Ybeta source. The sample was illuminated using 470 nm blue light for 50 seconds. Before the illumination, the sample presented 5 glow peaks, a prominent glow peak at 76 °C (I) and other glow peaks at115 (II), 180 (III), 290 (IV), 423°C (V). In the PTTL tests with blue light, it was possible toobserve that peak I presented the phenomenon of PTTL. Based on these tests, peak I worksas an electron acceptor and peaks II as the main electron donors for the observed PTTLsignal in peak I. In addition, peak I and II are the main components responsible for the observed rose quartz PTTL.

Acknowledgments:I. A. Ferreira thanks to FAPESP (#2021/05042-0, #2022/14925-5) and IFSP. M.C.S. Nunes thanks to FAPESP (#2021/12758-1, #2022/14516-8). N. M. Trindade is grateful to FAPESP (#2018/05982-0, #2019/05915-3), CNPq (#409338/2021-4).

[1] Chithambo, M.L., Folley, D.E., Chikwembani, S., 2019. Phototransferred thermoluminescence from natural quartz annealed at 1000°C: Analysis of time-dependent evolution of intensity and competition effects. J. Lumin. 216, 116730.

[2] Yukihara EG, McKeever SWS, Andersen CE, et al. Luminescence dosimetry. Nat Rev Methods Prim 2022; 2: 26.

Brazilian minerals (alexandrite, quartz and fluorapatite) as ionizing radiation detectors

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Recently we have studied some luminescent properties of alexandrite (BeAl2O4:Cr3+), quartz (SiO2), and fluorapatite [Ca10(PO4)6F2], using the thermoluminescence (TL) technique. TL corresponds to the light emission upon heating of insulating or semiconducting materials previously exposed to ionizing radiation, besides incandescence [1]. The TL signal was detected with a bialkali PMT behind a UV-transmitting, visible-absorbing glass filter (Hoya U-340, 7.5 mm thick). Irradiation was performed at room temperature using the built-in 90Sr/90Y beta source (dose rate of 10 mGy/s).

Alexandrite showed five TL glow peaks, I (~355K), II (~405K), III (~445K), IV (~530K), and V (~580K), at a heating rate of 1K/s. In this case, peaks IV and V were investigated because they present more useful characteristics for dosimetry. Alexandrite showed good linearity in the TL signal of both peaks as a function of dose (0.5 to 5 Gy). For rose quartz, TL analyses showed an intense glow peak in ~355K, at a heating rate of 1K/s. The TL dose response showed a weak sublinear behavior in the dose range from 0.01-0.1Gy and, after that, a supralinear tendency. In fluorapatite, the TL curve showed three glow peaks located about I (~350K), II (~470K), and III (~570 K), at a heating rate of 1K/s. The dose response, from 1 to 5 Gy, is linear for all TL glow peaks in fluorapatite.

With this work, we expect to have contributed with useful information regarding the use of luminescent properties of minerals for dosimetry purposes.

Acknowledgments: Matheus Nunes thanks to FAPESP (#2021/12758-1; 2022/14516-8); Isabela Ferreira thanks to FAPESP (#2021/05042-0; 2022/14925-5). Roberto Martins thanks to FAPESP (#2022/07200-4). Alexia Silva thanks to FAPESP (#2020/15626-6). Neilo Trindade and Elisabeth Yoshimura are grateful to FAPESP (#2018/05982-0; #2019/05915-3) and CNPq (#311657/2021-4; #409338/2021-4).

References:

[1] S.W.S. McKeever, Thermoluminescence of Solids, Cambridge University Press, Cambridge (1985).

Effects of radiation on the crystal lattice of minerals: a case study on Zircon and Apatite.

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In this work, micro-Raman spectroscopy characterization [1] of Apatite and Zircon is presented. These minerals are commonly used in geo-thermochronological dating methods such as Fission-Track Thermochronology (FTT), U-Pb and others. Both minerals have some ppm Uranium and Thorium content in their crystal lattices. In this context, the crystal lattice can feel the radiation effects when these atoms fission and/or decay. In view of this, it is important to investigate how these damages interfere with the mechanisms used in the application of FTT (e.g., diffusion kinetics, chemical attack and annealing). Thus, aliquots of these minerals were analyzed under different conditions: i) mineral under natural conditio ii) after total annealing; iii) post-irradiated with high energy ions, and; iv) post irradiation with high energy ions + alpha particles. These conditions are intended to simulate damage to the crystalline lattice by only 238U fission and/or metamictization (lattice destruction due to alpha decay). In zircon case, the spectra show that the irradiated samples (i.e.; ZIR and Ziα) suffered an apparent damage by reducing the intensity and FWHM increase at 1008. On the other hand, the restorative effect of heating to the crystal lattice is perceived by the sample ZAN that presents a greater intensity in relation to the ZST sample. Apatite shows that, at 963 cm-1, the irradiated samples (i.e.; AIRPR and AiαPR) show an increase in the intensity and narrowing of the FWHM. These intensity variations and FWHM indicate the beginning of a phase transition and/or increase of the amorphous phase in relation to the crystalline phase. New characterizations via SAXS and TEM may indicate in more detail how the crystal lattice responds to irradiation.

Acknowledgements:
CAPES and FAPESP (processes 2021/05660-5).

References:

[1] DIAS, A. N. C. et al. Journal of Raman Spectroscopy, v. 40, p. 101-106, (2009).

Fluorescence microscopy: Unfolding spatial distribution of color centers and metallic colloids inionic crystals irradiated with GeV ions?

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Investigating the radiation interaction with matter allows one to verify predictions based on theoretical modeling as well as to develop various applications, as those present in nuclear medicine and nanotechnology. When matter is in the solid state, ionizing radiation is able to transfer its energy to the target causing structural modifications. However, the mechanisms responsible for the conversion of the transferred energy to these structural modifications are still on debate in the literature and only a robust experimental database would furnish the means to unfold the most efficient mechanisms for materials with different physical and chemical properties. In this presentation, we address the formation of defects in calcium fluorite crystals irradiated with heavy ions in the electronic regime, focusing on the possible use of fluorescence microscopy. Due to the extremely large band gap, point defects are able to trap electrons and holes. These centers absorb electromagnetic radiation in the visible spectrum and fluoresce [1], providing information concerning the kind of defect, its concentration, and its spatial distribution. Due to the partial radiolysis of the crystals when exposed to radiation, extended defects are associated to metallic colloids and gaseous inclusions [2]. The colloids scatter electromagnetic radiation and induce surface enhanced Raman scattering, providing information concerning their size distribution [3]. We estimate the expected fluorescence signal and present preliminary results.

Acknowledgments:

Financial support from Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP, 2022/14906-0).

References:

[1] K. Schwartz, C. Trautmann, R. Neumann. Nuclear Instruments and Methods in Physics Research B 209, 73 (2003)

[2] R. Bennewitz, D. Smith, M. Reichling. Physical Review B 59, 8237 (1999)

[3] I. Alencar, J. Ruiz-Fuertes, K. Schwartz, C. Trautmann, L. Bayarjargal, E. Haussühl, B. Winkler. Journal of Raman Spectroscopy 47, 978 (2016)

Investigation of the Optically Stimulated Luminescence of Brazilian Alexandrite stimulated with different light sources

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The potential of natural alexandrite as a dosimeter was investigated because the chemical nature of this mineral, $BeAl_2O_4:Cr^{3+}$, combines the two oxides most commercially used for applications using optically stimulated luminescence (OSL), these being BeO and Al_2O_3. OSL occurs when an insulator or semiconductor is exposed to ionizing radiation and after an optical stimulus, the material emits photons in proportion to the radiation dose given previously [1]. The measurements were carried out using a Risø OSL reader (model DA-20). OSL emission was stimulated using different light-emitting diodes: blue (470 nm), green (525 nm), and infrared (850 nm), in constant wave (CW-OSL) and linear modulation (LM-OSL) mode. Irradiation was done using a built-in $^{90}Sr/^{90}Y$ beta source. The alexandrite powder samples showed a luminescent signal for the dose range from 0.1 to 1Gy for the three different types of LED. The CW-OSL and LM-OSL curves could be mathematically deconvolved into 3 components labeled fast, medium, and slow. The intensity of the curves and consequently of the components was more intense for stimulation with blue LEDs. The dose-response is linear and reproducible for the studied dose range. In general, alexandrite presents promising behavior for applications in the dosimetry field.

Acknowledgments:

M.C.S. Nunes thanks to FAPESP (#2021/12758-1; #2022/14516-8). E. M. Yoshimura is grateful to FAPESP (#2018/05982-0). N.M. Trindade thanks FAPESP (#2018/05982-0; #2019/05915-3) and CNPq (#409338/2021-4).

References:[1] E.G. Yukihara, S.W.S. Mckeever, Optically Stimulated Luminescence: Fundamentals and Applications, UK: John Wiley and Sons, West Sussex, 2011.

Kinetics of chemical etching in isotropic, solid-state nuclear track detectors: Predicting etch-pit diametersof alpha particles from a bulk source

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Radiation is essential to life. However, up-to-date it is still not possible to distinguish clearly the safe limit between benefic and malefic effects caused to organic tissue by exposition to ionizing radiation. Particularly, there seems to be an apparent contradiction in utilizing radiation sources for cancer therapy with their potential correlation as a factor responsible for cancer. In this way, it is mandatory to monitor radiation levels, especially in dwellings, where there is a low air circulation. For such purpose, high-resolution, active detectors are not well-suited due to the relatively low radiation activity present. On the contrary, passive nuclear track detectors, based on the processes of registration and revelation of the ionizing radiation trail, are more promising. Generally, these detectors are composed of low cost polymers, which allows a broad evaluation of the activity distribution in a whole geographic region. In order to identify the energy and mass of the nucleus responsible for the track formation, it is crucial to correlate these parameters with the observables after track revelation. Notably, etch-pit diameter is the simplest parameter to quantify and an extensive theoretical modeling has been proposed to its kinetic description [1]. Nevertheless, only the asymptotic behavior has been utilized in the literature. In this work, I present a reappraisal of previous results on alpha spectrometry in poly-allyl-diglycol-carbonate (PADC) using an americium source [2]. The Kolmogorov-Smirnov test is applied as the figure-of-merit for the etching kinetics and an independent prediction for irradiation with a bulk source is compared to experimental results.

References:

[1] G. Somogyi and S.A. Szalay, Nuclear Instruments and Methods 109, 211 (1973)

[2] C.J. Soares, I. Alencar, S. Guedes, R.H. Takizawa, B. Smilgys, J.C. Hadler, Radiation Measurements 50, 246 (2013)

Synthesis and thermoluminescence studies of Ce^{3+}doped calcium aluminate

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Calcium aluminate ($CaAl_2O_4$) has full potential to be designed for thermoluminescence (TL) applications like persistence luminescence and dosimetry [1]. Recent studies show that Ce^{3+} can be a good candidate in the $CaAl_2O_4$ matrix for TL applications. TL is a powerful technique used for dosimetry of ionizing radiations. Ce^{3+} doped $CaAl_2O_4$ was prepared by the combustion synthesis method. $Al(NO_3)_3$, $Ca(NO_3)_3$, and urea are used as starting materials. Urea acts as a fuel for combustion and $Ce(NO_3)_3$ provides the doping ions. The mixture of reagents was kept in a conventional furnace at 500ºC for about 5 min. After combustion, the crystalline powder was crushed in a porcelain crucible, then it was annealed at 950ºC for about 2 h. Phase formation was determined by X-ray diffraction (XRD) patterns. XRD pattern of Ce^{3+} doped $CaAl_2O_4$ was collected using an X-ray diffractometer (Panalytical - Empyrean) with a Cu-Kα (λ= 1.5 Å). The X-ray tube was operated at 45 kV and 30 mA. The 2θ degreeswere recorded from 0° to 50° in a 0.02° step. TL measurements were performed with theRisø reader (model DA-20) composed of a beta source $^{90}Sr/^{90}Y$ (dose rate 10mGy/s) used toirradiate the sample and a Hoya U-340 filter (transmission window 290-370 nm). The samplewas irradiated from 0.010 Gy to 1 Gy. The TL glow curves of the samples were deconvolutedusing the TGCD package. In addition, the dose response, reproducibility, and repeatabilitywere studied. XRD analysis showed high crystalline formation of the monoclinic phase. Thesamples show linear dose response of $R^2 = 0.9993$ in the range studied. So far, it can beconcluded from this preliminary study that $CaAl_2O_4:Ce^{3+}$detectors have potential for applications as dosimeters.

Acknowledgments:
W. R. S. Júnior thanks to CAPES. E. M. Yoshimura is grateful to FAPESP (#2018/05982-0). N. M. Trindade is grateful to FAPESP (#2018/05982-0, #2019/05915-3), CNPq (#409338/2021-4).

References:
[1] R.K. Gartia et al., Journal of Luminescence 219, 116867 (2020)

Whey-assisted proteic sol-gel method as a newsynthesis route for β-spodumene

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β-spodumene (or β-LiAlSi2O6) is a ceramic of great interest due to its optical properties, and has been evaluated for ionizing radiation dosimetry. The proteic sol-gel method has some advantages for the synthesis of this material, such as low synthesis temperatures, high homogeneity and low cost. Whey protein is a promising ligand for the method, as it is considered eco-friendly. Cheese whey represents an important environmental problem when discarded into water sources, because of the high volumes produced and its high organic matter content. The objective of this research is to evaluate the efficacy of the partially hydrolyzed whey protein as an eco-friendly ligand in the sol-gel method for the synthesis of β-spodumene. Structural characterization by X-ray diffraction (XRD) and morphological characterization by scanning electron microscopy (SEM) were performed. Rietveld refinement of X-ray powder patterns (XRPP’s) and mean crystallites size by Debye-Scherrer’s (D-S) and Williamson-Hall’s (W-H) methods were carried out. The XRPP’s and the Rietveld refinement fit confirmed the formation of the single phase β-spodumene for samples calcined at heat-treatment temperatures of 1000 °C and 1100 °C, compatible with reference ICSD 14235, with tetragonal crystal structure. D-S and W-H analysis concluded that the mean crystallites size tend to increase with the increase of the heat-treatment temperature, decreasing the microstrain, due to crystal coalescence. SEM analysis identified polycrystalline particles of micrometric order, with irregular and angular morphology. This research demonstrates the innovative efficacy of the whey as a ligand in the sol-gel method for β-spodumene synthesis, contributing to the perspective that wastes are progressively replaced by suitable treatments for this application.

Acknowledgements:
The authors are thankful to the CAPES and FINEP (ref. 0533/16).

CaF_2 detectors based on Brazilian natural fluorite: a preliminary study of radioluminescence for real time dosimetry

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CaF_2detectors (synthetic and natural forms) exhibit a broad range of linear dose responses to various types and energies of ionizing radiation with thermoluminescent (TL) and optically stimulated luminescence (OSL) techniques. The aim of the present work is to study the radioluminescent (RL) response of CaF_2detector based on greenish Brazilian natural fluorite (CaF_2:N) in diverse wavelengths. We produced CaF_2OSL film composed of CaF_2:N crystals in a semi-organic matrix and then made 6 mm diameter pellets. RL signals were evaluated using x-ray photons of 40kV of LUMI22 spectral TL reader (homemade at University of Sao Paulo). The luminescence emission spectrum was detected with an optic fiber spectrometer (Ocean Optics - model FLAME-S-XR1-ES) based on a CCD detector. The three CaF_2samples were irradiated three times with exposure times to the X-ray beam of 10 up to 60 seconds, the background measure was carried out with the same exposure times used to detect the RL, the measurements obtained were the absolute value of the glow intensity as a function of the detected wavelengths emitted. The dose-response analysis was performed from the mean of the three measurements subtracted from the background and by the integral of the dosimetric peaks. It was verified that the signals obtained in the first two peaks emitted in the UV and violet regions are proportional to the dose, and that the dose response is linear in the range of 7 mGy up to 450 mGy. Therefore, it can be concluded in this preliminary study that CaF_2:N detectors have potential for real-time dosimetric applications.

Acknowledgements:
M.C.S. Nunes thanks to FAPESP (#2021/12758-1; #2022/14516-8). N.M. Trindade thanks FAPESP (#2018/05982-0; #2019/05915-3) and CNPq (#409338/2021-4).

Characterization of optically stimulated luminescence (OSL) response of X-ray irradiated alexandrite-polymer composites

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The application of luminescence materials in the dosimetry field is based on the relation between the energy absorbed by ionizing radiation and the intensity of light emitted when the material is stimulated by light or heat [1]. If the luminescence is stimulated by optical energy, it is called optically stimulated luminescence (OSL). Natural and synthetic luminescent materials can be used as luminescent detectors. Alexandrite ($BeAl_2O_4$: Cr^{3+}) is a mineral with potential for OSL detector because its chemical structure is based on the combination of BeO and Al_2O_3, both commercially used as OSL dosimeters [2]. In this study, powdered alexandrite mixed with a polymeric matrix, in the form of pellets, were investigated for OSL dosimetry. The pellets were produced with 10 wt% of alexandrite powder (>75 μm) and 90 wt% of SIQMOL binder. The samples were irradiated with X-Rays using a Moxtek 50 kV cabled source. The OSL measurements were carried out using a Risø reader (model DA-20) with blue light emitting diodes (470 nm, FWHM = 20 nm) delivering 90 mW/cm^{2}at the sample in constant wave mode. R Studio software was used to perform the deconvolutions of OSL curves. Dose-response (0.035 - 0.284 Gy), repeatability and reproducibility were carried out to investigate the samples. The results demonstrate that alexandrite has a linear signal in function of exposition under X-Rays, with a correlation coefficient of R^2= 0.96, and the deconvolution of OSL curve presented three components: fast, medium, and slow. Repeatability test had a variation coefficient of ~ 4.6% (9 runs) and reproducibility of ~ 27.6% (9 samples) was obtained.

Acknowledgements:

Y.F. Amorim thanks to FAPESP (#2022/06690-8). M.C.S. Nunes thanks FAPESP (#2021/12758-1; #2022/14516-8). N.M. Trindade is grateful to FAPESP (#2018/05982-0; #2019/05915-3) and CNPq (#409338/2021-4).

References:

[1] P. Olko, Radiation Measurements 45, 506-511 (2010).

[2] V.Yu. Ivanov and V.A. Pustovarov et al, Phys Solid State 47, 466-473 (2005).

Charge-up and metallization effects in LiF crystalsduring sequential ToF-MeV-SIMS with continuous primary ion beams

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Ionic crystals do not amorphize when exposed to ionizing radiation. However, when a large amount of energy is deposited in the target (beyond its linear response), partial radiolysis may occur and its surface becomes metallized. For instance, irradiation of lithium fluoride (LiF) crystals with a beam of energetic ions (MeV range) causes fluorine diffusion, which may lead to fluorine bonding and formation of a gas bubble that could escape from the crystal surface. Meanwhile, due to the similarity to the cation substructure, lithium forms a metallic region. This metallization is a cumulative effect of radiation as a function of the total fluence of particles [1]. The initial motivation for this study was to investigate the metallization process of LiF crystals using Time-of-Flight Secondary Ion Mass Spectrometry with continuous, energetic primary ions (ToF-MeV-SIMS), yet another phenomenon was noted when performing the data analysis. An additional electric field, related to surface charging-up, was observed when bombarding the crystals with 6 MeV copper ions at a charge state of 5+. This field shortened the ToF of secondary ions ejected from the target to the detector. In this work, we determine this additional field and calculates the charge density at the surface, which is in accordance to the accumulation of charge from the primary ions, for the positive secondary ions of lithium and lithium oxide. As similar irradiation conditions are often employed for ion implantation, our findings shed light on reported flux-dependent observations [2].

Acknowledgements:

Financial support from Fundação de Amparo à Pesquisa e Inovação do Estado de Santa Catarina (FAPESC, public call 48/2021).

References:

[1] J.A.M. Pereira and E.F. da Silveira, Nuclear Instruments and Methods in Physics Research B 127,157 (1997)

[2] A. Lushchik,Ch. Lushchik, K. Schwartz, E. Vasil’chenko, R. Papaleo, M. Sorokin, A.E. Volkov, R.Neumann and C. Trautmann, Physical Review B 76, 054114 (2007)

CRYSTALLOGRAPHIC AND ISOTOPIC CHARACTERIZATION OF APATITE MINERAL OF PLUTONIC ROCKS FROM THE SÃO JOSÉ DO CAMPESTRE TECTONIC DOMAIN, NATAL, RN, BRAZIL

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Apatite can be characterized by observing the tiny etch tracks that become visible when it is placed in a dilute nitric acid solution at room temperature for about 20 seconds [1]. These tracks are produced cumulatively over the geological history of the mineral and their retention age can be determined through half-life data, the amount of ^{238}U, and the number of decays that have occurred [2]. This study aims to characterize apatite grains collected from three plutonic rocks in the northern part of the Borborema Province, Natal, RN, Brazil. Crystallographic characterization is performed using the Fission Track Method (FTM), micro-Raman spectroscopy, and scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). Isotopic characterization is done through the FTM using a Laser Ablation system coupled to a mass spectrometer (LA-ICPMS). Studying the physical-chemical phenomena that occur during the natural crystallization of apatite, a new protocol for its crystallographic characterization prior to determining its FT age can be developed. The Borborema Province is a region of great structural complexity in northeastern Brazil with several proposed models for its geological evolution [3]. Thus, with this work, the thermal history can be calculated based on the results, and a standard sample from Brazil can be determined to calculate the age of samples collected from other regions of geological interest.

Acknowledgments: This work was supported by FAPESP (Process No2022/07601-9).

References:
[1] Malusà et al. Fission-Track Thermochronology and its Applications to Geology. 2019.
[2] Donelick et al. Apatite fission track analysis. Reviews in Mineralogy and Geochemistry, 58, 49-94, 2005.
[3] Caxito et al. Toward an integrated model of geological evolution for NE Brazil-NW Africa: The Borborema Province and its connections to the Trans-Saharan (Benino-Nigerian and Tuareg shields) and Central African orogens. Brazilian Journal of Geology, 50(2). 2020.

CRYSTALLOGRAPHIC AND ISOTOPIC CHARACTERIZATION OF THE ZIRCON MINERALPEIXE, TOCANTINS, BRAZIL, TO USE AS AGE STANDARD FOR THE FISSION TRACK METHOD

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Zircon is a common accessory mineral in igneous, metamorphic and sedimentary rocks, which is able to incorporate trace elements of 5 to 4000 ppm U and 2 to 2000 ppm Th [1]. This makes it widely used in dating processes. This work aims to characterize and date zircon megacrysts from Peixe Alkaline Complex, Brazil, via Raman spectroscopy, optical microscopy, scanning electron microscopy, mass spectrometry LA-ICP-MS and Fission Track Method (FTM), in order to classify this zircon sample and determine whether it can be used as an age standard for the FTM, as a Brazilian sample. The studied zircon mineral from Peixe are megacrystals with dimensions in the order of centimeters that occur in the nepheline syenites of the complex [2]. Because of that, we will cut it into its different faces in order to observe whether the track density has anisotropic behavior. We will do the same by reducing part of the megacrystal into grains (length around 200 µm) with random crystallographic orientations. By using FTM with LA-ICP-MS, it will be possible to obtain simultaneously the fission track age and the isotopic characterization of this sample, that could serve as a standard. It is believed that the zircon megacrystals from Peixe Alkaline Complex nepheline syenites crystallized at 1503±3 Ma, and suffered a metamorphic-metasomatic event that led to Pb loss at about 577 Ma [2].

Acknowledgements:
This work was supported by FAPESP (process n° 2022/11589-4).

References:
[1] H. Gorz. Chemie der Erde, vol.33, 326-357 (1974).
[2] L.F.W. Kitajima, J. Ruiz, G. Gehrels, and J. C. Gaspar. In Simposio Sul Americano de Geologia de Isotopos, vol.3. 2001.

Crystals Growth Ce-doped BaZrO3 Scintillators

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Investigations of BaZrO3 Perovskite compounds suggest that the band-gap and its trappings are largely responsible for luminescence emissions, and their shape can be controlled by the conditions of synthesis of nanocrystals by the Microwave-Assisted Hydrothermal Method (MAH) [1-2]. Crystals doped with lanthanides have received considerable attention due to the allowed 5d-4f transition, such as trivalent cerium (Ce3+) with good light yield and energy resolution achieved in many materials, having intrinsic phenomena such as luminescence or fluorescence by conversion energy [3]. This work aims to investigate the properties of BaZrO3 Perovskite polycrystals doped with Cerium (Ce) Lanthanides grown by the MAH method. BaZrO3 samples were synthed by the MAH method and characterized using XRD and SEM. XRD results indicated the majority crystalline phase of BaZrO3 with a cubic lattice. SEM images show the present shape and regular from undoped samples. BaZrO3 samples will be synthed and doped with Ce, with the purpose of allowing the identification of changes in electronic structures, their influence on the synthesis process, establishing correlations with their optical properties and investigating energy conversion processes compared to pure BaZrO3, to be analyzed by means of photoluminescence, fluorescence, X-ray absorption and decay measurements.

Acknowledgements: CAPES, CNPQ, FAPERGS, UFPEL, CCAF, UFS and PPGFIS.

[1] M. L. Moreira et al. Scripta Materialia 64, 118-121 (2011).
[2] R.U. Fassbender et al. Phys. Chem. Chem. Phys., 17, 11341-11349 (2015).
[3]R.U. Fassbender et al. Journal of Electron Spectroscopy and Related Phenomena 251, 147106 (2021).

Effect of heat treatment on the optical, structural, and magnetic properties of natural kyanite samples

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Kyanite (Al_2SiO_5) belongs to the aluminum silicate class of materials, with their composition mainly attributed to the Al_2O_3 and SiO_2 structural units [1,2]. The mineral's color varies due to impurities, such as Cr, Ti, V, Mn, and Fe. Kyanite is widely used in developing ceramics materials due to its excellent thermal properties and has been investigated as an ionizing radiation detector. This work investigates the optical, structural, and magnetic properties of blue and green kyanite samples from Minas Gerais, submitted to thermal treatment at 300, 500, 700, 900, and 1100°C. The x-ray diffractograms confirmed the triclinic crystalline structure of the studied samples. The energy dispersive spectroscopy (EDS) results show that the samples mainly comprise silicon, oxygen, aluminum, and iron. X-ray fluorescence data reveal the presence of some impurities, including Iron (Fe), Vanadium(V), Titanium (Ti), Chromium (Cr), and Manganese (Mn). The absorption peaks observed forthe samples can be attributed to the substitution of Al^{3+} by Fe^{3+} ions and the Ti^{4+}-Fe^{2+}charge transfer. The magnetic behavior of the as-prepared samples and those submitted to athermal treatment of 1100 ºC was investigated at 2 K and 300 K temperatures. Electronparamagnetic resonance (EPR) spectra revealed the presence of iron ions in the crystallinestructure of the green and blue kyanite samples, with larger spectral line widths for thegreen kyanite, indicating a higher iron content than for the blue sample. X-ray fluorescencestudies also showed higher iron contents in green kyanite than in the blue sample, confirming the EPR results.

Acknowledgments: This work was supported by CNPq and FAPESP (#2021/13132-9).

References:

[1] P. BOCH and J.C. NIÈPCE, Ceramic materials: processes, properties and applications, Wiley-ISTE (2007).

[2] A. Tarantola, P. Voudouris, A. Eglinger, C. Scheffer, K. Trebus, M. Bitte, B. Rondeau, C. Mavrogonatos, I. Graham, M. Etienne, et al. Minerals 9, 252 (2019).

Fission-tracks dating in apatite: provenance studies by LA-ICP-MS

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Apatite is a phosphate mineral, rich in uranium and present in various types of igneous, metamorphic and sedimentary rocks. This mineral is the most used by Fission-Tracks dating (FT), allowing the reconstruction of thermal events that occurred in the first kilometers of the upper crust, in addition to its use in sedimentary provenance studies. FT dating is based on the quantitative relationship between the density of parent and daughter isotopes and their decay constant. To estimate the number of daughter atoms, the FT dating method is based on the accumulation of defects generated by the spontaneous fission of 238U in the mineral´s crystalline structure. The closing temperature for apatite is around 120 °C, from which the record of FT formation is preserved [1]. The amount of parent atoms (238U), can be discovered using Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS). This technique estimates the amount of 238U of grain directly and quickly [2]. The methodological procedures used in this work consist of choosing the apatites, mounting grains in epoxy resin, polishing, and subsequent etching with nitric acid to reveal the FT. Subsequently, for each grain, the surface FT are counted in an optical microscope and is measured confined FT and etch pit, used as a kinetic parameter (Dpar) to realize the thermal modelling of the sample. The next step is to mensure 238U directly in the mineral, using LA-ICP-MS. The different ages of fission tracks obtained in studies of sedimentary provenance allow differentiating the sources of sediments.

Acknowledgments:This work was supported by FAPESP.

References:

[1] Laslett, G.M.; Green, P.F.; Duddy, I.R.; Gleadow, A.J.W. Chemical Geology, 65 (1), 1-3 (1987).

[2] Soares, C.J.; Guedes, S.; Hadler, J.C.; Mertz-Kraus, R.; Zack, T.; Iunes, P.J. Physics and Chemistry of Minerals, 41, 65-73 (2014).

INVERTED MODEL OF ANNEALING VIA AGES OF FISSION TRACKS BY LA-ICP-MS OF APATHITE SAMPLES COLLECTED FROM THE PERUVIAN CENTRAL ANDES.

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Under an adequate chemical etching, the tracks left by the nuclear fissions of 238U are enlarged and can be seen under an optical microscope to determine the density of fossil tracks. 238Uppm can be evaluated using the LA-ICP-MS spectrometry technique by relating the average counts per second of the isotopes of 238U and 43Ca with the 238Uppm of a known standard sample. With the density of fossil tracks and the 238Uppm it is possible to calculate the corrected age of the sample based on the shortening of confined tracks produced by the annealing process depending on the temperature and time conditions these samples are subjected in their place of origin. The variety in the lengths of confined tracks can be represented by a length probability density function that characterizes their distribution. Then, from the Monte Carlo method, thermal histories are artificially obtained with a probability density function of confined tracks lengths and apparent ages. These thermal histories can be discarded or appreciated as the observed data and theoretical data converge towards statistical compatibility. Two samples of apatite were submitted to the inverted model to determine their thermal histories with the purpose of proposing a geological model of tectonic dynamism, taking into account what is already known about the region ([1]Chavez et al., 2021; [2]Mamani et al., 2010).

This work was supported by FAPESP and CNPq.

References:

[1]CHAVEZ MACHACA, C. A., RODDAZ, M., DANTAS, E. L., SANTOS, R. V., ALVÁN, A. A. Provenance of the Middle Jurassic-Cretaceous sedimentary rocks of the Arequipa Basin (South Peru) and implications for the geodynamic evolution of the Central Andes. Gondwana Research, 101, 59-76 (2021)

[2]MAMANI, M., WÖRNER, G., SEMPERE, T. Geochemical variations in igneous rocks of the Central Andean orocline (13 S to 18 S): Tracing crustal thickening and magma generation through time and space. Bulletin, 122(1-2), 162-182 (2010)

INVESTIGATION OF ALPHA PARTICLE EJECTIONFROM ZIRCON MINERAL

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Zircon contains traces of unstable actinide elements [1], whose radioactive decay comprises the emission of alpha, beta and gamma radiation particles in the chains (U-Pb and Th-Pb) and the spontaneous fission events of the U-238 isotope. The self-irradiation process, in geological time, damages the zircon crystalline structure by altering the physical and chemical properties of the bulk mineral. Alpha decay is the radioactive event that contributes the most to bulk damage to the mineral lattice and the loss of radiogenic Pb and He, which results in skewing of geological ages.The refore, a quantitative assessment of alpha emanation from the zircon surface was performedusing a CR-39 as an external alpha detector. A CR-39 detector in 2π geometry exposed to a zircongrain for 60 hours at a temperature of 40° degrees resulted in an average alpha-track density of 10^4 tracks/cm 2 . Moreover, an investigation of the alpha emanation from different zircon grains from standard samples (FCT and KGD) indicates the inhomogeneity of U and Th concentrations in the minerals, which may disperse the measured U-Th/He ages. The heterogeneous emanation of particles can be attributed to imperfections in the crystalline structure that may be inherent to the crystallization of zircon or acquired during its geological history [2]. Understanding the alpha emission of zircon in relation to its crystalline structure is fundamental for the U-Pb and (U-Th)/He methods.

Acknowledgements: This work was supported by FAPESP (Process N o 2020/15144-1).

References:

[1] Ahrens, L.H., Cherry, R.D., Erlank, A.J., 1967. Observations on the Th-U relationship in zircons from granitic rocks and from kimberlites. Geochimica et Cosmochimica Acta. 31, 2379-2387.

[2] Resende, R.S., Tello, S., C.A., Pereira, L.A.S., Dantas, E.L., 2019. The effect of chemical and physical imperfections in zircon grains in influencing the U-Pb age analyses: Insights from zircon fission track etching. Lithos, v. 346-347.

Photodetection in bismuth chalcohalides pellets: purevs composite materials

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Bismuth based semiconductor materials are increasing in popularity due to their potential in optoelectronic applications such as solar cells and radiation detection. In particular, BiSI has been studied for these purposes, especially in film deposition. In this work, we presentthe study of photodetection in BiSI pellets. BiSI nanorods were synthed by either a solutionor solvothermal method, both developed in our group [1]. The solution method yielded pure crystalline BiSI nanorods in the Pnma crystal group, while the solvothermal method produced a composite of BiSI nanorods and amorphous carbon particles. The pellets were constructed by cold pressing in a uniaxial press, followed by a heat treatment for 3.5 hours at 250ºC under Ar atmosphere. Two pellet thicknesses were tested: 1.5 and 2.5 mm, while the diameter was kept at 10 mm. Pellets made with the pure product showed proved to be more brittle than pellets with the composite. Prototype devices were built by depositing Au contacts through evaporation and Pd wires were attached with Aquadag and encapsulated with acrylic.

I-V curves were measured both in the dark and under UV light. Measurements were carried out up to 600 V and the current measured with a picoammeter. The resistivity was increased four orders of magnitude from $10^8$to $10^{12}$when changing from the pure material to the composite, respectively. This is in accordance to the fact that the composite has amorphous structure that limits the conductivity. However, the response to UV light was similar in both cases, ranging from 0.5-0.6. This work is a first approximation of a simple photodetection device with an easy to synthe novel material.

Acknowledgments: Prof. Mauricio Rodríguez Chialanza for cold press

[1]Mombrú Frutos, M. et al. Understanding the Crystal Growth of Bismuth Chalcohalide Nanorods through a Self-Sacrificing Template Process: A Comprehensive Study. Inorganic Chemistry 61, 9231-9241 (2022).

Production of Transparent Ceramics Based on YAG: Ce ($Y_{3-x}Ce_xAl_5O_{12}$) for Use as X-rays Detectors Using Colloidal Process

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Scintillators play a central role in modern engineering as converters of high-energy ionizing radiation (such as X-rays) into visible light, being one of the main components of radiation detectors. To meet the high demand for these components, in combination with the development of new materials and techniques for the production of increasingly efficient scintillators, a series of research has been carried out in recent decades. Yttrium Aluminum Garnet (YAG) is a material that stands out in different fields of scintillation. To produce transparent ceramics scintillators, in this work, Ce-doped YAG nanoparticles were prepared by the co-precipitation method, followed by calcination at 1100 °C. To achieve the desired microstructure and transparency for the final application of the material, conformation, and sintering were tailored to achieve the best parameters for obtaining such properties. Seeking to achieve a high green density through a colloidal process, using the pressure slip casting method, the concentration of solids in the colloidal suspension was a key parameter, as well as the colloidal stability and pressure applied during the slip casting. The final densities obtained for the different parameters were measured and compared. In this way, the purpose of this work is to present a methodology for the production of transparent ceramics scintillator by pressure slip casting method, seeking to reach the final properties necessary for use in the Sirius fourth generation Synchrotron, located at the National Center for Research in Energy and Materials (CNPEM) in Brazil.

Radiation Damage in Zircon Grains: A NewClassification Based on Optical Analysis

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Zircon is a widely-used accessory mineral that is valuable in the study of geothermal and chronological events, providing insights into the geological history of the Earth. Due to its crystallographic properties, this mineral has the ability to accumulate heavy chemical elements, such as uranium in its crystal lattice at concentrations in parts per million (ppm) [1]. Zircon is also capable of detecting self-radiation, making it a useful tool in this field of study. The 238U isotope spontaneously fissions, leading to the release of high energy and the creation of a disturbance in the crystal structure of nanometric dimensions known as fission track. This phenomenon provides valuable structural information about zircon and can also be used to date the mineral using the fission-track method (FTM) and U-Pb chronometry [2]. After etching, tracks can be observed on zircon surface under an optical microscope. The appearance of these tracks can vary depending on the structural characteristics of the grain. Some layers may not reveal fission tracks or may be destroyed, while others may remain intact or display different patterns of track density. These variations allow the mineral to be classified into different categories [3], with distinct patterns observed depending on the properties of the parent rock, such as whether it is from a new or older region, granitic, or from a basin. Based on the optical analysis of over 1000 zircon grains from various samples, a new classification of zircon grains is proposed.

Acknowledgements:This work was supported by CNPq.

References:

[1] H. Gorz. *Chemie der Erde*, 33, 326-357 (1974)

[2] R. S. Resende, S. C. A. Tello, E. L. Dantas, P. Hackspacher, M. C. A. Cháves, U. A. Glasmacher. *Journal of South American Earth Sciences*, p. 102-512. (2020).

[3] M. C. Azevedo. Dissertation (Master in Materials Science). *Universidade Estadual Paulista*. (2021).

Radioluminescence of Ce^{3+} doped $CaAl_2O_4$ as a function of annealing temperature

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Recent studies show that Ce^{3+} can be a good candidate in the $CaAl_2O_4$ matrix for scintillation purpose. The scintillators are luminescent materials that absorb ionizing radiation and convert it into light within the region of the visible/IR. These devices have applications such as dosimetry, nuclear medicine and high energy physics. To measure the scintillation properties, radioluminescence (RL) is a powerful technique used since RL is a prompt luminescence emitted by a material under ionizing radiation exposure [1]. In this work, undoped and cerium-doped ($CaAl_2O_4$:Ce^{3+}) powders were synthesized by combustion synthesis in solution. Aluminum nitrate, calcium nitrate, and urea are used as starting materials. Urea acts as a fuel for combustion and cerium nitrate provides the doping ions. The mixture of reagents was kept in a conventional furnace at 500ºC for about 5 min. After combustion, the crystalline powder was crushed in a porcelain crucible. The samples were annealed for 2h at five different temperatures (600, 700, 800, 900, and 1000°C); and the effects of ionizing radiation were investigated using the RL technique. To irradiate the samples, we used a custom-made equipment named LUMI22, that consists of an X-ray tube (Moxtek 50kV, 0.2mA), a spectrometer (Ocean Optics, FLAME-S-XR1-ES, range 200-1050nm) coupled with a fiber optic (QP1000- 2-UV-VIS). Preliminary results of the undoped sample showed a broad RL peak at ~520 nm which can be caused by intrinsic defects, e.g., oxygen vacancies. Doped samples showed a peak at 400 nm which may be caused by 5d-4f transition in the crystalline structure. So far, it can be concluded from this preliminary study that $CaAl_2O_4$:Ce^{3+} detectors have potential for applications as scintillators.

Acknowledgments:

W. R. S. Júnior thanks to CAPES. N. M. Trindade is grateful to FAPESP (#2018/05982-0, #2019/05915-3), CNPq (#409338/2021-4).

References:

[1] L.V.S. França, L.C. Oliveira, O. Baffa, Measurement 134, 492-499 (2019)

Response of CsI:Pb scintillator crystal to neutron radiation

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The helium-3 world crisis requires a development of new methods of neutron detection to replace commonly used ^{3}He proportional counters. In the past decades, great effort was made to developed efficient and fast scintillators to detect radiation.[1] These detectors should, then, be carefully characterized both experimentally and by means of advanced simulation code. Ideally, the detector should have the capability to separate neutron and gamma induced events either by amplitude or through pulse shape differences. As neutron sources also generate gamma radiation, which can interfere with the measurement, it is necessary that the detector be able to discriminate the presence of such radiation. Considerable progress has been achieved to develop new inorganic scintillators, in particular increasing the light output and decreasing the decay time by optimized doping.

Crystals may be found to suit neutron detection. In this report, we will present the results of the study of lead doped cesium iodide crystals (CsI:Pb) grown in our laboratory, using the vertical Bridgman technique. The concentration of the lead doping element (Pb) was studied in the range $5x10^{-4}$M to 10^{-2}M. The crystals grown were subjected to annealing (heat treatment). In this procedure, vacuum of 10^{-6} mbar and continuous temperature of 350°C, for 24 hours, were employed. In response to neutron radiation, an AmBe source with energy range of 1 MeV to 12 MeV was used. The activity of the AmBe source was 1Ci Am. The fluency was 2.6×10^6 neutrons/second. The operating voltage of the photomultiplier tube was 1300 V; the accumulation time in the counting process was 600 s and 1800 s. The scintillator crystals used were cut with dimensions of 20 mm diameter and 10 mm height. The Monte Carlo method was used to determine the neutron flux arriving in the detector and the calculated values were obtained by means of MCNP code.

References

[1] G. F. Knoll, Radiation Detection, 4th edition,2010.

Study of optically stimulated luminescence propertiesof amazonite

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In the group of K-rich Feldspars, amazonite is a variety that attracts attention due to its blue-greenish color. Recently, efforts have been made to understand and apply the luminescence properties of this mineral [1]. In view of that, the aim of this work was to study the Optically Stimulated Luminescence (OSL) of amazonite, a microcline feldspar variety (KAlSi3O8). OSL characteristics, such as dose-response, repeatability and reproducibility, and fading were analyzed in a TL/OSL commercial reader Risø DA-20. The samples were irradiated with a beta source 90Sr/90Y, delivering a dose rate of 10 mGy/s. Also, chemical and structural characterization have been done from X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD), respectively. The amazonite showed the presence of some impurities, such as PbO, one of the agents in the coloration of amazonite; and, from XRD data, it was possible to observe an albite phase. The CW-OSL curves showed a high intensity with a fast decay for the dose range studied (0.01 - 0.3 Gy), and from the coefficient of linearity g(D), it was possible to conclude the dose-response showed a linear behavior for the range studied. For reproducibility and repeatability, a coefficient of variation (C.V.) of 3.7% (12 samples) and 0.4% (12 runs), respectively, was obtained. OSL fading at room temperature was ~23% within one hour after irradiation.

Acknowledgements: R.T.E.K. Martins thanks to FAPESP (#2022/07200-4). E.M. Yoshimura is grateful to CNPq and FAPESP (#2018/05982). N.M. Trindade is grateful to FAPESP (#2018/05982-0, #2019/05915-3), CNPq (#409338/2021-4).

References:

[1] Sfampa. I.K, Polymeris. G. S, Pagonis. V, Theodosoglou. E,Tsirliganis. N. C, Kitis. G, Journal of Nuclear Instruments and Methods in Physics Research B, 2015

Thermoluminescence, Fluorescence and Electron Paramagnetic Resonance of natural alexandrite.

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Recent studies have proposed the investigation of alexandrite mineral ($BeAl_2O_4:Cr^{3+}$) as a potential luminescent dosimeter. Despite this, in-depth studies of defects and luminescent centers in alexandrite were not investigated. It is proposed to characterize the thermoluminescence (TL) emission spectra, fluorescence (FL) and electron paramagnetic resonance (EPR) of alexandrite. TL dosimeters, when stimulated by thermal energy, emit light proportional to the dose of ionizing radiation previously absorbed. The emitted wavelength can be measured through thermoluminescence emission spectra. On the other hand, EPR and FL provides additional information about mineral structure, enabling the study of the effects of ionizing radiation on the sample. For TL, the samples were irradiatedwith X-rays and measured by Lumi22 homemade system. EPR measurements were carriedout using a Bruker EMXplus spectrometer operating in the X-band at room temperature, 2mW microwave power, 100 kHz modulation frequency and 0.25 mT modulation amplitude. The FL spectra were measured using the Duetta spectrofluorometer, with a 75 W xenon arclamp included. The data was collected by scanning excitation wavelengths in 360-480 nmand recording emission in the wavelength range of 670-740 nm. Preliminary results indicatethat the TL emission spectra presented two peaks, at ~80°C and ~175°C, with emissioncentered between 600 nm - 700 nm, related to Fe^{3+} and Cr^{3+}, also detected by EPR. TheFL spectra showed two signals: an emission centered at 680 nm (Cr^{3+} in inversion center),and at 700 nm (Cr^{3+} in mirror symmetry). The results of all techniques used in this worksuggest that emissions are correlated to Cr^{3+} and Fe^{3+} ions.

Acknowledgements: A. O. Silva thanks to FAPESP (#2020/15626-6). M. C. S. Nunes thanks to FAPESP (#2021/12758-1). E. M. Yoshimura is grateful to FAPESP (#2018/05982-0). N. M. Trindade thanks FAPESP (#2018/05982-0; #2019/05915-3), CNPq (#409338/2021-4) and IFSP/IFSULDEMINAS.

M-Topology in Quantum Physics

Disentangling edge and bulk contributions in spin pumping of YIG/MoS$_2$ triangular flakes

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Recently, spintronics has emerged as a candidate to supplant conventional silicon-based electronics. A notable milestone in spintronics was achieved by Dyakonov and Perel, who identified the transverse accumulation of spin in correlation to a charge current[1], called the Spin Hall Effect. Also, it is essential to consider that these moments can originate fromeither the spin moment or orbital moment through the orbital Hall effect (OHE). In addition,this accumulation of spins can also be related to other effects, such as the Valley Hall effect(VHE) and the Rashba-Edelstein effect (REE). High-order topological materials known as transition metal dichalcogenides (TMDs), particularly the semiconductor MoS2, have gained significant interest among the materials that exhibit these distinct effects. This material predicts OHE, VHE, and REE for states near the Fermi energy. The existence of SHE is only expected for the conducting states of this material. Therefore, it is possible to induce the SHE through the electronic population of these conducting states. This population of the conducting states can be obtained by multiple approaches, such as by exciting the valence band electrons with light or by the presence of conductive edge states in specific symmetries of this material [2,3]. Spin pumping is an experiment that investigates specific effects. It injects angular momentum dynamically from a nearby ferromagnetic layer. A magnetic insulator like YIG can reduce the impact of specific effects and create a pure spin current. We focused on evaluating the contributions of different effects that generate magnetic moment accumulations transverse to the charge currents, specifically looking at the induced spin Hall effect.[1] D’ Yakonov Perel J. Exp. Theor. Phys. Lett. 3023, 1971. [2] T. Cysne et al. PRL. 126.5 (2021), 056601. [3] S. H. Safeer et al. ACS. Appl. NanoMat 4.4 (2021), 4172-4180. [4] R. Torrão et al. J of All. Comp. 923 (2022), 166300.

Emergent phenomena from topology in tellurium

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Elemental tellurium crystalizes in covalently bound atomic helices, arranged in a hexagonal lattice in which the helices are connected via the van-der-Waals interaction. It is a semiconductor with a 330 meV band gap and a strong spin-orbit interaction which leads to a unique spin texture of the valence band. Along the helical screw direction, the spin texture allows parallel or antiparallel current-induced magnetization and implies a recently identified spin Hedgehog, when samples are sufficiently p-doped [1]. We present an experimental AC-magneto transport study for different frequencies of Sb-doped and chemically pure needle-shaped tellurium single crystals. The current flows along the chiral screw axis, while the direction of the external magnetic field is perpendicular and longitudinal in relation to the chiral screw axis. The behavior of resistance as a function of frequency is consistent with topological effects which includes the emergence of a strong pseudomagnetic field.

[1] G. P. Maruggi, J. Ferreira, E. Baggio-Saitovitch, C. Enderlein, e M. B. S. Neto, “Hedgehog orbital texture in p -type tellurium and the antisymmetric nonreciprocal Hall response”, Physical Review Materials, vol. 7, no 1, jan. 2023, doi: 10.1103/physrevmaterials.7.014204.

Machine learning for materials discovery: Two-dimensional topological insulators

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One of the main goals and challenges of materials discovery is to find the best candidates for each interest property or application. Machine learning rises in this context to efficiently optimize this search, exploring the immense materials space, consisting of simultaneously the atomic, compositional, and structural spaces. Topological insulators, presenting symmetry-protected metallic edge states, are a promising class of materials for different applications. However, further development is limited by the scarcity of viable candidates. Here we present and discuss machine learning-accelerated strategies for searching the materials space for two-dimensional topological materials. We show the importance of detailed investigations of each machine learning component, leading to different results. Using recently created databases containing thousands of ab initio calculations of 2D materials, we train machine learning models capable of determining the electronic topology of materials, with an accuracy of over 90%. We can then generate and screen thousands of novel materials, efficiently predicting their topological character without the need for a priori structural knowledge. We discover 56 non-trivial materials, of which 17 are novel insulating candidates for further investigation, for which we corroborate their topological properties with density functional theory calculations. This strategy is 10× more efficient than the trial-and-error approach while a few orders of magnitude faster and is a proof ofconcept for guiding improved materials discovery search strategies.
[Schleder et al. Appl. Phys. Rev. 8, 031409 (2021)]

Multiple strong topological gaps and hexagonalwarping in Bi_4Te_3

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The electronic topology of Bi_4Te_3, composed of alternating $Bi_2$and Bi_2Te_3layers, was investigated by density functional theory and by angle-resolved photoemission spectroscopy (ARPES). We find, remarkably, that there are three adjacent strong topological gaps with associated protected surface states within a 2-eV range of the Fermi level. Additionally, a surface-state Fermi surface with strong hexagonal warping is observed. The spin texture of topological states in Bi_2Se_3around the Fermi level shows a linear dispersion with all spins states parallel to the surface with no warping shaped Fermi surfce[1], unlike, the $Bi_2Te_3$and Bi_4Te_3that show a hexagonal warping[2] of the electronic states in the vicinity of the Fermi level. In the Fermi surface with this sort of warping the spin texture has a component out of plane of the surface[3]. As in $Bi_4Te_3$the warping was found to be much stronger then in Bi_2Te_3, it shell have a larger out of plane spin component which impplies in a high potential applications in spintronics. In this presentation I will show the experimental results for $Bi_4Te_3$and I will also discuss how the combination of ARPES technique and synchrotron radiation helped to solve this system.

References:

[1] K. Kuroda, M. Arita, K. Miyamoto, M. Ye, J. Jiang, A. Kimura,E. E. Krasovskii, E. V. Chulkov, H. Iwasawa, T. Okuda, K.Shimada, Y. Ueda, H. Namatame, and M. Taniguchi, HexagonallyDeformed Fermi Surface of the 3D Topological InsulatorBi2Se3, Phys. Rev. Lett. 105, 076802 (2010).
[2] Y. L. Chen, J. G. Analytis, J.-H. Chu, Z. K. Liu, S.-K. Mo, X.L. Qi, H. J. Zhang, D. H. Lu, X. Dai, Z. Fang, S. C. Zhang, I. R.Fisher, Z. Hussain, and Z.-X. Shen, Experimental realization ofa three-dimensional topological insulator, Bi2Te3, Science 325,178 (2009).
[3] L. Fu, Hexagonal Warping Effects in the Surface States ofthe Topological Insulator Bi2Te3, Phys. Rev. Lett. 103, 266801(2009).

The “ABC” of adatomic collapse in correlated Moiréless multi-graphene

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In this talk, we discuss the multi-graphene of ABC-type stacking, namely, the 2D version of the 3D multi-Weyl semimetals with the topological charge Fano effect[1], and its possibility of having virtual bound states, in particular, residing within the Coulomb insulating gap of an Anderson-like adatom[2]. Wondrously, one of the virtual states constitutes the counterpart of the atomic collapse phenomenon proposed in relativistic atomic Physics, while the second emerges as its particle-hole symmetric, analogous to a positron state. Thus, the effect is introduced as the adatomic collapse, which occurs due to a flat band with a dispersionless state and a divergent density of states $\sim|\varepsilon-\varepsilon_F|^{2/J-1}$ for $J\geq3$ near the Fermi energy ε_F, where $J\pi$ is the Berry phase. We verify this scenario based on the Kramers-Kronig transformation of the quasiparticle broadening, from where we observe that the aforementioned van Hove singularity induces virtual bound states. Counterintuitively, near the singularity, we reveal these states above and below the Fermi energy correlated to the existence of the bottom and top edges of the Coulomb insulating region, respectively. As such a behavior rises without a twist, the system is known as Moiréless and the phenomenon emerges also assisted by the adatom Coulomb correlations. Therefore, the current findings make explicit that the ABC multi-graphenen system is a rich environment for strongly correlated phenomena and emulation of relativistic Physics.

[1] W.C. Silva, W.N. Mizobata, J.E. Sanches, L.S. Ricco, I.A. Shelykh, M. de Souza, M.S. Figueira, E.Vernek, and A.C. Seridonio, Phys. Rev. B 105, 235135 (2022); [2] W.C. Silva, A.M. Freitas, L.T. Lustosa, J.E. Sanches, M. de Souza, and A. C. Seridonio, arXiv:2304.02680 (2023).

ACKNOWLEDGMENTS: CNPq Grant. Nr. 308695/2021-6; FAPESP Grant Nr. 2018/09413-0.

Unveiling quantum phase transition by disorder and defects in 2D-materials: Jacutingaite Family

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Transition metal dichalcogenides have been the subject of numerous studies addressing technological applications and fundamental issues. Within this class of materials, a single-layer PtSe2 is a semiconductor with a trivial bandgap. In contrast, its counterpart with 25% of Se atoms substituted by Hg, Pt2HgSe3 (jacutingaite, a naturally occurring mineral), is a 2D topological insulator with a large bandgap. We investigate the energetic stability and the topological transition in Pt(HgxSe1-x)2 as a function of alloy concentration and the distribution of Hg atoms embedded within the PtSe2 host. Our findings reveal the dependence of the topological phase on the alloy concentration and robustness regarding the distribution of Hg in ordered or random configurations. We also show that vacancies randomly distributed induce a quantum transition trivial to topological in doped transition metal dichalcogenides. Finally, we discuss a fundamental issue, whether a topological insulator protected by time-reversal is robust enough under a transformation to an amorphous state. The crucial role of translational symmetries in building the theory of topological insulators raises the question: to what extent is translational symmetry necessary for a topological state to retain its properties?

N-Processing, Properties, Modelling and Characterization of Metals and Alloys

Compositional and cycling effects in several high entropy alloys foreseen for solid-state hydrogen storage

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Among various materials for solid-state hydrogen storage, alloys and intermetallics forming hydrides are one of the most important classes due to their high-volume density, reversibility, and safety. Recently, the metallurgy paradigm of high entropy alloys (HEAs), initially intended to enhance the mechanical properties [1], has been proposed for hydrogen storage [2]. The principle is laid on the mixing of elements close to the equimolar proportion for systems up to five and more elements. This may lead to the formation of simple single-phased solid solutions (bcc, fcc and hcp) with interesting properties for solid-sate hydrogen storage. We will focus here on the physicochemical and the hydrogen absorption/desorption properties of several series of HEAs (containing mainly refractory, transition and light metals) and the insights that can be obtained from experiments at large-scale facilities, mainly neutron and X-ray diffraction [3-5]. The role of chemical composition will be highlighted as function of the local lattice distortion and electronic properties [3-5]. For example, in situ neutron diffraction at D1B ILL has clearly evidenced a drastic decrease of the desorption temperature associated with the phase transition from a fcc hydride to a bcc alloy in the Al-containing materials [6]. Moreover, the absorption/desorption cycling effects on these materials will be clarified by the help of advanced characterisation techniques such as, pair distribution function analysis of total X-ray scattering and positron annihilation spectroscopy. This approach is envisioned to clarify the role of the chemical composition and the cycling effect on the hydrogen storage performances of HEAs.

[1] D.B. Miracle, O.N. Senkov, Acta Mater. 122 (2017) 448-511.
[2] M. Sahlberg et al., Sci. Rep. 6 (2016) 36770.
[3] A. Bouzidi el al., Hydrogen. 2 (2021)
[4] N. Pineda-Romero, C. Zlotea, Materials. 15 (2022) 7974.
[5] C. Zlotea et al., Front. Energy Res. 10 (2022).

Corrosion behavior of high entropy alloys

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In this study, the effects of three different nickel concentrations on the microstructure, hardness, and corrosion properties of AlCrFeCoNix (x = 1.0, 1.5, 2.0) high entropy alloys (HEA) were investigated.

The alloys were obtained by arc melting from high-purity raw materials and subjected to heat treatment at 1125 ◦C for 5 h. Microstructural characterization revealed a dendritic morphology for the equimolar and AlCrFeCoNi1.5 alloys, while the AlCrFeCoNi2 alloy presented a hypoeutectic microstructure. Differences in microstructure and chemical composition were observed between the dendritic and interdendritic regions, with clear segregation of Cr in the interdendritic areas. The increase in Ni content and the heat treatment resulted in the formation of the FCC phase, consequently reducing the alloys' hardness.

The corrosion properties of HEA were evaluated by potentiodynamic polarization and electrochemical impedance spectroscopy methods in a 3.5% NaCl solution. A passive film was formed at the corrosion potential for the three compositions, that resulted in high resistance to localized corrosion mainly after heat treatment as indicated by higher pitting potential and larger passive plateau. EIS results indicate that the protective film's formation depends on Ni content, which is also related to the FCC/BCC ratio and phase compositions. The heat treatment resulted in a more homogeneous and thicker oxide film, as indicated by the reduction of Q and Ceff of the alloys in this condition. The protective film's formation depends on Ni content, which is also related to FCC/BCC ratio and phase compositions.

Design, Processing and Hydrogen Storage Properties of Multicomponent Alloys Derived from the TiZrNbCrFeNi System

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Multicomponent alloys have been considered as a new class of metal hydrides. The extensive compositional field that can be explored opens new horizons to find novel metal hydrides with tunable hydrogen storage properties [1,2]. In this study, two multicomponent alloys with a single C14 laves phase and a VEC of 6.4 were designed by thermodynamic calculations from the TiZrNbCrFeNi system with different A/B ratios, such as (TiZrNb)60(CrFeNi)40 and (TiZrNb)40(CrFeNi)60. Our aim was to investigate the influence of A and B elements (A: elements forming hydride, B: elements with low chemical affinity with hydrogen) of the TiZrNbCrFeNi system on the hydrogen storage capacity, activation, absorption, and desorption kinetics. We prepared the selected alloys by arc melting under an inert atmosphere and analyzed their microstructure using XRD with the Rietveld method, SEM/EDS, and TEM characterization techniques. The hydrogen storage performance was evaluated using absorption/desorption kinetics and PCT isotherms. Our results revealed that the arc-melted equiatomic TiZrNbCrFeNi alloy contained two C14 Laves phases with slight differences in unit cell parameters, while the non-equiatomic alloys had a high amount of C14 laves phase with minor traces of BCC phases with fractions inferior to 13%. The microstructural features demonstrated the existence of compositional gradients in the alloys, which matched very well with the different phases observed and fitted in the XRD analysis. At 473 K, the equiatomic TiZrNbCrFeNi alloy could reversibly absorb and desorb 1.1 wt% of hydrogen with a maximum H/M ratio of 0.75. The fractions of A and B elements in the TiZrNbCrFeNi alloy directly impacted the performance on activation, hydrogen capacity, and kinetics of the alloys studied here.

Developments and Industrializations of Zr-based Bulk Metallic Glasses and Fe-based Glassy and Nanocrystalline Alloys

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Multicomponent alloys have attracted increasing interest for the last three decades since the first synthesis of multicomponent bulk metallic glasses (BMGs) in 1990. The multicomponent alloys reported to date are classified to BMGs, BMG composites, high entropy (HE) BMGs, HE crystalline alloys and marginable BMGs. When we focus on engineering application, the most widely commercialized alloys are BMGs. Their BMGs are roughly classified to nonmagnetic Zr-based and ferromagnetic Fe-based type compositions. The former type is typically composed of Zr-Al-Ni-Cu and Zr-Al-Ni-Cu-(Ti,Nb) systems and the latter type is Fe-Cr-(P,B,C,Si), Fe-(Cr,Nb)-P-B and Fe-(Cr,Nb)-(P,B,Si) systems. The commercialization products have been usually produced by direct die casting from liquid for the Zr-based BMGs, while the Fe-based glass-type alloys have been produced by high-pressure gas atomization and ultrahigh water atomization. The former BMGs have been used as various structural materials such as casing, housing, pin spring, hinge, ratch cover writing tool, precise gears, knife, optical mirror, sporting goods and ornaments etc., while the latter glass-type alloys exhibit unique soft magnetic properties with the features of low coercive force (Hc), good high-frequency permeability, low core loss and high electrical resistivity in combination with high glass-forming ability which can be prepared as spherical powders over the whole particle range by high-pressure gas or ultrahigh-pressure water atomization and the powder-consolidated cores have been used as high performance inductors with low core losses even in a high frequency range up to 3 MHz. The low core loss characteristics combined with high-frequency permeability have attracted rapidly increasing engineering importance, because of the applications to advanced articles such as smartphone, notebook PC, DC/DC convertor, point of load power supply, digital camera, automobile AV equipment, car navigation system and RFID sheet etc. New

Effect of Solution Heat Treatment on Microstructure and Properties of Ni-15Nb-M alloys (M = Al, Ti, Cr, Fe)

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Ni-based superalloys are widely used in critical components of aircraft engines and turbines and also in the petrochemical industry, for applications in highly corrosive environments [1]. These alloys have as main characteristics their superior mechanical, corrosion and oxidation resistance at high temperatures, as well as creep resistance. The chemical composition associated with carrying out heat treatments directly influences the phases formed (such as the ordered cubic phase γ'-$Ni_3(Al,Ti)$ in the fcc γ-Ni matrix), and depending on the alloying elements and fraction, there is the possibility of an increase in mechanical strength through hardness. There is a certain gap in the literature regarding the study of ternary Ni-Nb-based superalloys [2], and the influence of the third alloying element on the microstructure and properties. In this context, the objective of the study is to characterize pseudo-eutectic alloys of the Ni-15Nb-M system (M = 2 to 15 wt.% for Al, Ti, Fe, Cr) and investigate the influence of alloy elements and solution heat treating (SHT) on their microstructure and properties through X-Ray Diffraction (XRD), Optical Microscopy (OM), Scanning (SEM) and Transmission Electron Microscopy (TEM) and Vickers Microhardness. Thus, the main results are that the addition of Cr significantly affects the hardness and in the Ni-15Nb-Msamples the solution heat treatment showed less significant effect. All Ni-15Nb-M ternaryalloys system (M=Al, Ti, Cr, Fe) pseudo-eutectic alloys showed microhardness and microstructural changes after solution heat treatment. The alloying elements that most contributed to increase the mechanical strength of the as-cast Ni-15Nb alloy were Cr and Al and the Cr addition provided the highest hardness among all compositions. After solution HT, hardness of Ni-15Nb-M was significantly decreased in all samples, mostly in the Ni-15Nb-2Al and Ni-15Nb-15Cr.

Effects of warm-rolling process on CrMnFeCoNi multicomponent alloy

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The study aimed to investigate the impact of warm-rolling on the microstructure and mechanical properties of the equiatomic CrMnFeCoNi high entropy alloy (HEA) with a face-centered cubic (FCC) structure. Warm-rolling was performed on the HEA, achieving reductions in thickness of 76% and 90% at a temperature of 773 K. To elucidate the influence of warm-rolling, a comparison was made with cold-rolled HEA samples, both deformed with and without subsequent annealing. Despite undergoing similar deformation as the cold-rolled samples, they exhibited a lamellar microstructure that was notably coarser than the cold-rolled material. This disparity in microstructure could be attributed tothe dynamic annihilation of dislocations during the warm-rolling process. While the cold-rolled material displayed a single-phase FCC structure without any indication of second phases, it is essential to consider the presence of a Cr-rich body-centered cubic (BCC) phasereported in the literature during annealing at 773 K. Consequently, the possibility of a Cr-rich BCC phase should not be disregarded. Nano-twins were introduced during both warmand cold-rolling processes and were retained in the cold-rolled sample even after annealingat 773 K for 1 hour. Chemical fluctuations observed in the warm-rolled HEA indicated theformation of second phases, which was not observed in the similarly cold-rolled samples. Thus, warm and cold-rolling processes not only lead to distinct microstructures but also result in different local variations in composition. The warm-rolling may be a way to improvethe combination of strength and ductility compared to the cold-rolled counterparts. In summary, the findings of this study suggest that warm-rolling can be effectively employedas a thermo-mechanical approach for tailoring the microstructure and mechanical properties of the CrMnFeCoNi alloy, as well as other single-phase FCC high entropy alloys(HEAs).

Fapesp 2021/04302-8

High entropy alloys with fixed fcc-structure forhydrogen storage

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Alloys with at least five principal elements with atomic concentrations between 5 and 35% are called high entropy alloys (HEAs). This mixing often lead to the formation of simple single-phased solid solutions with body centered cubic structures. During hydrogenation, a phase transformation from the bcc to the fcc structure (face centered cubic) is often observed, with an increase of the unit cell and a volume expansion of the material in the macroscopic state. This swelling process can lead to various problems in the design of tank systems for solid metal hydrides. One possible option to prevent this process is to fix HEAs in the fcc-structure so that significant volume expansion cannot occur during hydrogenation. The synthesis and properties of fcc-fixed HEAs are demonstrated using various examples.

Hydrogen induced failure in Ti6Al4V0.1Ru alloy

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The challenge of exploring oil in extreme conditions, which includes the presence of gaseous CO2 and H2S and a deepwater salt layer, has led the material design of metallic components towards increasingly special alloys. In addition to high mechanical and corrosion resistance, these alloys must exhibit the resistance to hydrogen embrittlement caused in service. Hydrogen embrittlement is a major industrial problem, as it can occur in several metal alloys that are mechanically and corrosion resistant, such as nickel alloys (I718, I725), duplex, superduplex steels, among others. In this scenario of numerous attempts to search for alloys adapted to aggressive environments, titanium alloys of high specific mechanical strength and fatigue resistance, have been explored. It is noteworthy that titanium alloys still exhibit low density (d= 4.7g/cm3) when compared to Ni base alloys (d= 8.9 g/cm3) and Fe (d= 7.8 g/cm3). To achieve high mechanical properties, alloying elements should be added to titanium so that alloys with biphasic structure (α = HC and β=BCC) are obtained. Ti and Ti-based alloys form hydrides with hydrogen and become brittle. Thus, components made of these materials are coated with elastomer to avoid hydrogen embrittlement. In case of any rupture in this layer, the alloy may be in contact with hydrogen sources and be subjected to hydrogen embrittlement. In this context the present work aims to evaluate the hydrogen permeation and thermal desorption spectroscopy, together mechanical properties in the samples hydrogenated to elucidate the mechanism of degradation induced by hydrogen in Ti6Al4V0.1Ru.

Materials costumization by In-situ synthesis ofadvanced alloys

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A major contribution to a sustainable manufacturing chain is offer by a deep knowledge of the tripod processing/microstructure/performance. High efficiency of materials usage and an understanding of high performance materials and parts fabrication is a major step to a sustainable industry. Enhanced performance can be achieved with costumized materials for each application leading to longer service life a reduced industrial waste, particularly for parts operating under harsh conditions, such as high temperature applications where multiple phenomena act on the material simultaneously. In-situ synthesis of low weldability alloys to process protective coatings or parts by additive manufacturing during the deposition of powder mixtures presents adequate features to costumize parts. A competitive route to costumize materials is summarized and discussed, particularly for the in-situ processing of intermetallic alloys. The solidification of intermetallic processed by plasma transferred arc as single layer and multilayer deposits and the impact of the interaction with the substrate is discussed for nickel aluminides well known for their advanced performance. The impact of the interaction with the substrate in the ordered alloy that accounts for a higher hardness and it effect on high temperature wear is shown. Compositional and property gradients obtained from in-situ synthesis during multilayer processing allowing to process sound materials with either smooth or sharp gradients. Examples of carbide gradient in an aluminide matrix and the simultaneously synthesis of aluminide and ceramic layers are analyzed and their impact on wear performance discussed. Highlights from recent developments on the synthesis of Nb silicides and the challenge of in-situ processing multi-element compositions are discussed regarding its impact on a wider use of Nb alloys.

Mechanical deformations as a way to enhance hydrogen storage properties of metal hydrides

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Metal hydrides are attractive materials for a large range of hydrogen storage applications. However, a few characteristics have to be improved in order to make them more commercially suitable. In this talk we will review various mechanical deformation techniques that could enhance the hydrogen storage properties of metal hydrides. Cold rolling (CR), Equal channel Angular Pressing (ECAP), High-Pressure Torsion (HPT), and Ball Milling (BM) will be covered. The characteristics of each of these techniques will be discussed. Different examples of these techniques on a wide range of metal hydrides (Mg-based, LaNi5, TiFe, BCC alloys and High Entropy Alloys (HEA) will be shown.

Metal-based nanoparticles supported on date seed-activated carbon as electrocatalysts for the conversion of biomass derivatives into biofuels

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The conversion of biomass derivatives into biofuels through electrocatalysis is an effective and environmentally friendly method. To achieve selective production of biofuels, it is crucial to develop eco-friendly, robust, and sustainable electrocatalysts. This presentation showcases the results of nanoparticles deposited on date seed activated carbon supports, prepared and characterized using various analytical methods. The catalyst structure was analyzed through SEM,EDX, and X-ray diffraction, revealing that the metallic nanoparticles were successfully incorporatedinto the date seed activated carbon's disordered graphite-like structures. By simultaneously electrochemically hydrogenating (ECH) and oxidizing (ECO) furfural-based compounds, valuable biofuels such as furfural alcohol were obtained from biomass conversion. The electrocatalytic activity of the different catalysts was determined through cyclic voltammetry and electrochemicalimpedance spectroscopy. Catalysts were used with varying metal and content ratios to adjust furfural's ECH and ECO, resulting in different product yields and selectivity while also reducing the competing hydrogen evolution reaction. This study highlights the potential of converting biomassderivatives into biofuels to produce renewable energy.

References:

[1] M. A. Sabri, G. Bharath, A. Hai, M. Abu Haija, R. P. Nogueira, F. Banat, Fuel Processing Technology, vol. 238, p. 107525 (2022)
[2] L. Zhang, T. U. Rao, J. Wang et al., Fuel Processing Technology, vol. 226, p. 107097 (2022)
[3] W. Xu, C. Yu, J. Chen and Z. Liu, Applied Catalysis B: Environmetnal, vol. 305, p. 121062 (2022)

Microstructure and mechanical behavior of directionally solidified AlCoCrFeNi$_{2.1}$ and Al$_{0.8}$CrFeNi$_{2.2}$eutectic high entropy alloys

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One of the main motivations for carrying out eutectic growth study is the possibility of developing in-situ composite materials with special features, which provides an opportunity of merging the properties of distinct constituents into one material. The exciting microstructures found in several eutectic alloys result from cooperative growth, which is connected to the atomic transfer in the melt ahead of the solid/liquid interface. The features of intermetallic NiAl compound, with a B2 structure and a relatively low density and high oxidation resistance, melting temperature, hardness, and chemical stability make it an attractive material for the replacement of Ni superalloys. However, like many other intermetallic compounds, it shows poor ductility and low fracture toughness at room temperature. A strategy to address its lack of ductility while maintaining its mechanical strength is to combine it with another phase in the form of composite materials. The high-entropy alloys (HEAs) offer a new perspective to produce in-situ composites that are made up of brittle NiAl and a ductile phase, in which both phases are produced simultaneously during the solidification of a eutectic HEA (EHEA). In this context, this work explores the growth mechanisms of the two EHEA, AlCoCrFeNi2.1 (L ↔ NiAl-rich+ CoCrFeNi-rich) and Al0.8CrFeNi2.2 (L ↔ NiAl-rich+CrFeNi-rich) using directional solidification. The directionally solidified samples of both compositions exhibited a well-aligned microstructure with a lamellar morphology, regardless of the growth rate employed. The mechanical behavior of both EHEAs was assessed by compressive and Vickers hardness tests, and the results suggested that they exhibited remarkable mechanical behavior.

Acknowledgments:
We acknowledge the São Paulo Research Foundation, FAPESP, grant # 2018/18293-8, for financial support and LNNano (CNPEM/MCTI) for access to the SEM facilities.

Preparation of Highly Reactive Mg_2Ni Alloys and its Surface Protection Using Polyetherimide

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Hydrogen is an abundant and a promising energy carrier for cleaner energy applications. Therefore, solid-state hydrogen storage technologies using nanostructured materials is an important field of study. High gravimetric capacity and low cost make Magnesium-based alloys promising hydrogen storage materials, especially for stationary or electrochemical applications. Mg_2Ni has been extensively studied because of its relatively low enthalpy of formation. For the Mg_2Ni - based alloys, fast absorption kinetics at around 300 °C are usually obtained through complex routes, such as hydriding combustion synthesis or production of nanoparticles. In this study, we obtained Mg_2NiH_4 hydrides by reactive milling, annealing heat treatments and hydrogen absorption/desorption cycles at 350°C. Through this processing route, we obtained highly reactive hydrogen storage Mg_2Ni alloys, able to absorb and desorb hydrogen at temperatures lower than 300 °C, with desorption kinetics similar to those of alloys obtained by nanoparticle production or hydriding combustion synthesis and even better optimized absorption capacity. For example, a Mg_2Ni sample obtained by one of the routes investigated here showed hydrogen absorption capacity of 3.0 wt.% at 250 °C, with 2.5 wt.% absorbed in less than 50 seconds. A relevant surface protection effect of polyetherimine (PEI) introduced by solution mixing was confirmed for samples air-exposed for 01 day, 01 week and 01 month. Funding by FAPESP (The São Paulo Research Foundation) through process number 2019/20273-8 is greatly acknowledged.

QUick x-ray Absorption spectroscopy for TIme and space-resolved experiments: Opportunities for researchin energy materials

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In this talk, we will present the opportunities offered by QUATI beamline (QUick x-ray Absorption spectroscopy for TIme and space-resolved experiments) to research energy materials. The beamline is under construction at the 4th generation Brazilian synchrotron source Sirius [1]. QUATI will be dedicated to high-quality hard X-ray absorption spectroscopy for time and space resolved experiments under in situ/operando conditions. Thanks to the high intensity of photons and low emittance obtained from 4th generation facilities of synchrotron, fast acquisitions are expected, which are crucial for the study of dynamic processes.

The beamline’s design has focused on a high flux of photons in a wide energy range, 4.5-35keV (i.e. allows XAS from Ti to Xe in K edge other higher Z elements in L edges), with flexibility in beam (10’s μm to mm) and with focus towards a combination of multi-techniques. Taking advantage of the high flux of hard X-rays, it will be possible to perform not only conventional XAS measurements, but also spectro-tomography, high resolution -XAS and X-ray emission spectroscopy with dedicated instrumentations. Besides, a complete samples infrastructure is proposed to perform true operando characterizations with the combination of techniques, such as Raman, Infrared and X-ray diffraction. Finally, special attention has been dedicated to developing new software tools for the treatment of a large amount of data with multivariate analysis.

Acknowledgment FAPESP grant 22/01351-0

[1] L. Liu, N. Milas, A. H. C. Mukai, X. R. Resende and F. H. de Sá, The Sirius project, J. Synchrotron Rad. (2014). 21, 904-911. (https://www.lnls.cnpem.br/sirius-en/)

Solidification Behavior of Metallic Alloys by Selective LASER Melting

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Additive Manufacturing is an emerging industrial process based on selective laser melting of metallic alloys. Due to the very fast cooling rates (10^5 K/s) prevailing during solidification the microstructure presents micro/nanosegregation at cellular/dendritic boundaries and supersaturation of the matrix. The distribution of the alloying elements in this particular microstructure presents very special features that must be tailored to optimize the properties of the components. In this talk some results for AlSiMg-Fe alloy and Duplex Stainless steel are presented in order to highlight the magnitude of segregation , correlations with the estimated solidification rates and the effect of postprocessing heat treatments on the microstructure and properties

Acknowledgements: To FAPESP Thematic Project 2016/11309-0: ESTUDO, DESENVOLVIMENTO EAPLICAÇÃO DE PROCESSO HÍBRIDO: Manufatura Aditiva (MA) + High Speed Machining/Grinding (HSM/G) for the financial support

The estimation of the surface plastic deformation produced by grit-blasting in maraging steel using the strain-induced γ-α' transformation phenomena

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Grit-blasting is a treatment used in many industrial applications for cleaning and preparing metallic surfaces. Plastic deformation is induced by the high-velocity abrasive particles on the blasted metal surface, which may change mechanical properties in service. The quantification of such induced plastic strain is a difficult task, as the deformation mechanism involved in grit-blasting is complex. In quenched maraging steels, overaging leads to the transformation of martensite into reverted γ-austenite, which can be mechanically re-transformed into martensite by deformation at room temperature. This phenomenon can be used to estimate the plastic strain induced by grit-blasting. In the present study, overaged 18Ni(300) maraging steel samples were blasted at two different blasting angles (45º and 75º), keeping all other variables controlled at constant levels. The kinetics of γ-austenite into α′-martensite transformation was treated as a function of the strain, according to the Ludwigson-Berger model, parametrized by an experimental constant (Kp), related to the mechanical stability of the reverted γ-austenite and obtained from controlled uniaxial stretching testing at controlled deformation levels. The increase on the dislocation density (ρ) resulting from blasting could also be determined, by the Williamson-Hall method. The volume fraction of γ-austenite (fγ) was quantified in both blasted and stretched samples by ex-situ XRD analyses. It was found that α′-martensite was formed in blasted and stretched samples due to plastic deformation, after certain stress levels. Differences in strain rate between blasted and stretched samples and the consequent adiabatic heating effect on the kinetics of α′-martensite transformation were discussed. The true surface plastic strain induced by grit-blasting was estimated at 0.031 and 0.047 for blasting angles of 45º and 75º, respectively.

Advanced ferrous alloys and coatings for demanding applications

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Ferrous alloys are the first choice among metallic materials given a myriad of possibilities to design its structure in different length-scales to meet strict requirements. It is notorious the importance of steels on the success of developing modern societies. Iron (Fe) and its alloys are essential for improving the quality of life and enabling affordable and mass use technologies. Strength and toughness are vital requirements for most structural applications but are often mutually exclusive. Steel metallurgy proved to surpass this conflict through technologies at a reasonable cost, and research over the decades has enabled unprecedented levels of mechanical properties, including damage tolerance. Besides the economic and structural aspects, wear and corrosion resistance are also basic attributes of alloys aiming to withstand aggressive environments. In some cases, having corrosion and wear resistance is the first criterion for materials selection. This work summarizes the progress of advanced ferrous alloys and coatings in terms of alloy design, powder production, protective coatings, and how the microstructural features dictate the basket of properties, with focus on the tribological and electrochemical aspects.

Acknowledments: FAPESP (Grant No. 21/08514-0) and CNPq (Universal Grant No. 407651/ 2021-7).

Anomalous temperature-dependent behavior of thermophysical properties of CrFeNi, CoCrNi, and CoCrFeNi medium- and high-entropy alloys

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The linear coefficient of thermal expansion and specific heat capacity at a constant pressure of medium-entropy CrFeNi and CoCrNi alloys, as well as CoCrFeNi high-entropy alloy, showed an abnormal behavior in the temperature range 700-950 K. Magnetic moment measurements as a function of temperature demonstrated that the observed discontinuities are unrelated to the Curie temperature. The structural and microstructural alterations caused by heat treatments were studied in samples with varying thermal histories. According to transmission electron microscopy (TEM) and synchrotron X-ray diffraction data, second-phase precipitation, recrystallization, and texture are also not to blame. X-ray absorption fine structure shows that local lattice distortions are insignificant and that Cr and Ni elements have an unusual interaction. Combining the results of near-edge X-ray absorption fine structure and X-ray photoelectron spectroscopy indicates a pseudo-gap valley, which means a decrease in the overall Density of States at the Fermi energy upon alloying, with Cr and Ni being the primary contributors. The observed anomalies in thermophysical characteristics are attributed to chemical short-range order, also known as the K-state effect.

Acknowledgments: AFA acknowledges the financial support as a Postdoctoral fellow from the São Paulo Research Foundation (FAPESP), grant 2021/12056-7.

A protocol for making X-ray microtomography availablein an open laboratory scheme

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The Renato Archer Information Technology Center, through its Coordination of Technological Park and Open Laboratories (COLAB), will make available to Brazilian researchers a recently acquired Bruker/Skyscan 1272 CMOS X-ray microtomography equipment. Our first initiative was to perform a six-month pilot project aimed at creating a service protocol adapted to COLAB rules from project proposal submission to results delivery. A large variety of samples with different shapes, chemical compositions, and sizes were employed, including teeth, insect parts, microelectronic boards, 3D printed polymer, ceramic and metal parts, metal powder, and composite materials. The Bruker proprietary software for reconstruction (NRecon), visualization (CTVol, CTVox), and analysis (CTan) were utilized. The FileSender@RNP public interface was tested for sending large data archives. Additionally, the free software InVesalius, developed by CTI, was tested for providing users with basic measurements, segmentation, and visualization tools. We also tested remote access to software installed in CTI workstations for advanced reconstruction and measurements that require high computational processing. A preliminary protocol states that remote access to CTI workstations can be done satisfactorily but it is necessary to increase the computational capabilities of the CTI, even for a small number of users, to store the large amount of data that is generated. FileSender@RNP public interface worked well to tranfer large archives to the computer users. Data security measures should restrict access to users' data only. Concerning the samples, the metal ones must be small and thin, and those with low density should have transmission rates above 20%. Finally, the CTAn analysis is the user´s responsibility. Operating the X-ray microtomography equipment requires a 48-hour in-person training course.Acknowledgments:FINEP and CNPq for financial support References:[1] Bruker website , Skyscan 1272

Chromium carbides dissolution and corrosive attack on the surface of an AISI 430 stainless steel after heat treatment in different temperatures

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Non-stabilized stainless steel AISI 430 presents chromium carbides in its microstructure that are dispersed in the ferritic matrix of the steel. Depending on the thermal cycle used in the steel, these carbides preferentially precipitate in grain boundaries [1], generating a region pour in chromium next to them, what is a sensitization process[2]. These sensitizedregions are more susceptible to corrosive attacks [3]. This way, chromium carbides dissolution in AISI 430 had been evaluated after heat treatments in different temperaturesas well as what was the influence on the material's surface microstructure after corrosiveattack. After an initial characterization, hot rolled and annealed samples had been heat treated in an electrical furnace. Temperatures varied from 750 to 1050°C, in 80s cycles andwith fast cooling. From 950°C on, there is a decrease in the amount of carbides as the temperature of heat treatment increases. From 1000°C on, there is an increase in materialhardness and martensite is present in the microstructure. The heat treated samples hadbeen attacked with sulfuric acid and it was observed that the higher the heat treatment temperature, the smaller was the corrosive attack.

References:

[1] Krauss, G. Steels : Processing, Structure, and Performance. (ASM International,2015).

[2] Sarkari Khorrami, M., Mostafaei, M. A., Pouraliakbar, H. Kokabi, A. H. Studyon microstructure and mechanical characteristics of low-carbon steel and ferriticstainless steel joints. Mater. Sci. Eng. A 608, 35-45 (2014).

[3] Tavares, S. S. M., de Souza, L. F. G., Chuvas, T. de C. Machado, C. L. da C.Influence of heat treatments on the microstructure and degree of sensitization ofbase metal and weld of aisi 430 stainless steel. Rev. Mater. 22, (2017)

Design of a Ti-V-Nb-Cr alloy with hydrogen storage reversibility at room temperature

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Implementing hydrogen as an energy carrier to decarbonize industry applications might be very challenging. In this context, metal hydrides are a suitable option for solid state storage applications. Recently, Ti-V-Nb-Cr body-centered-cubic (BCC) alloys have shown promising storage properties including the ability to absorb hydrogen without the need of an activation treatment. However, the obtention of a Ti-V-Nb-Cr BCC alloy that absorbs/desorbs hydrogen at moderate pressure/temperature conditions with easy activation and cycling stability remains a challenge. In the present work, we demonstrate an approach to design a Ti-V-Nb-Cr BCC alloy with hydrogen absorption/desorption reversibility at room temperature and moderate pressure. CALPHAD (Calculation of Phase Diagrams) method and a thermodynamic model [1] to calculate pressure-composition-temperature (PCT) diagrams were employed to find compositions prone to form BCC alloys with plateau pressures above 1 bar at room temperature. Three BCC Ti-V-Nb-Cr alloys were synthesized via arc melting and showed the capacity to absorb hydrogen with fast kinetic and without activation treatments. PCT diagrams demonstrated reasonable agreement with the predicted diagrams calculated via modelling. Moreover, one of the designed alloys (Ti11V30Nb28Cr31) showed the capacity to reversibly absorb/desorb ~1.7 wt.% of H at room temperature and moderate pressure conditions. These results are unprecedent for the Ti-V-Nb-Cr system and emphasize the importance of alloy design strategies via computational tools.

Acknowledgements:

Serrapilheira Institute (Serra-1709-17362), FAPESP (2020/07707-6), CNPq (Grant number: 309467/2021-7 and 407906/2022-3). This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001, Federal University of Sao Carlos, Graduate Program in Materials Science and Engineering.

References:

[1] O. A. Pedroso et al., IJHE 47,32582-32593 (2022)

Effect of annealing temperature on the final size ofNiSi2 and CoSi2 nanoplates endotaxially grown in Si(100) wafers covered by Ni and Co salts

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The nanocomposites formed by crystalline $NiSi_2$ and $CoSi_2$nanocrystals embedded in silicon wafers have attracted the attention of many researches due to the potential of its application in nanotechnology. In this study we describe the effect of thermal treatment temperatures on the final size of well oriented $NiSi_2$and $CoSi_2$nanoplates grown in Si(001) wafers in a thin layer close to its external surface. The preparation of the silicide nanocrystals consisted in the deposition of a solution of Ni and Co salts on bare Si wafers, to obtain the $NiSi_2$and $CoSi_2$nanoplates, respectively, followed by thermal treatments of the samples under H_2flow at temperatures favorable to the diffusion of the Ni and Co atoms from the thin film into Si wafers and subsequent reaction with Si atoms. It was observed that this preparation method results in the formation of $NiSi_2$and $CoSi_2$silicide nanoplates with the shape of nearly regular hexagons having their larger surfaces parallel to one of the planes of Si{111} crystallographic form [1,2]. Samples on which salts of Ni or Co were deposited were annealed at different temperatures in the 340-700 °C range during 1 h. The average thickness and lateral side of the hexagonal silicide nanoplates formed in Si were determined from the analysis of grazing-incidence small-angle X-ray scattering (GISAXS) measurements performed after the thermal treatments.

Acknowledgements:

This work was made possible through the financial support of CAPES and CNPQ.

References:

[1] D. da Silva Costa et al., Surfaces and Interfaces, 29, 101763 (2022).

[2] G. Kellermann et al., Phys. Chem. Chem. Phys.,100, 063116 (2012).

Effect of C and N addition on the grain refinement and mechanical behavior of the $Cr_{40}Co_{40}Ni_{20}$ Multi-Principal Element Alloy

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Medium/high entropy, Multiprincipal element alloys (MPEAs) have significantly increased the number of potential compositions of metallic alloys to unprecedented levels. In additionto discovering new compositions of interest, optimizing the most promising MPEAs is equally critical. The $Cr_{40}Co_{40}Ni_{20}$MPEA stands out by showing a high mechanical strengthening via grain refining, and high work hardening capacity, resulting from the strain-induced HCP phase formation (TRIP effect) [1]. An important optimization route is the use of interstitial elements as microstructural refiners [2,3], which should be especially advantageous in alloys that have high sensitivity of mechanical properties in relation to grain size, such as CrCoNi MPEAs. Therefore, the objective of this work is to study the effect of interstitial elements (C and N) on the phase equilibrium, grain refinement and mechanical behavior of $Cr_{40}Co_{40}Ni_{20}$MPEA. The base alloy, two compositions with N, and two with C were selected by the CALPHAD method, produced with high purity elements, and annealed at four different temperatures. Microstructural analysis and hardness tests were carried out. The interstitial elements, either in solid solution or in the form of carbides and nitrides, had an influence on phase stability, recrystallization onset, grain refinement and hardness. In-situ tensile tests using synchrotron radiation were performed on some samples. The influence of interstitials on mechanical properties, stacking fault energy and deformation mechanisms are discussed.

Acknowledgements:
FAPESP for financial support Grant 2021-05408-4. This study was financed in part by CAPES - Finance Code 001.

References:
[1] Bertoli,G. et al. Applied Physics Letters,v.119,p.61903,2021.
[2] Klimova,M. et al. Journal of Alloys and Compounds,v.851,p.156839,2021.
[3] Jodi,D.E. et al. Materials Characterization,v.157,p.109888,2019.

Exploring the Compositional Flexibility and Mechanical Properties of Multi-Principal Element Alloys

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Multi-Principal Element Alloys (MPEAs) are alloys that do not have a single principal element, and therefore encompass an immense number of possible compositions. In terms of mechanical behavior, some compositions with a range of interesting properties have been discovered. Notable compositions reported include those of the Cr-Co-Ni, Fe-Mn-Cr-Ni, and Co-Ni-V systems, which are among the toughest materials ever developed. This high toughness can be attributed to the various hardening and deformation mechanisms of this material when subjected to mechanical stress. The fact that these alloys have a very large compositional flexibility makes it impractical to propose new compositions by trial-and-error methods. In this work, a series of results will be presented that have been carried out by our research group to efficiently explore this large compositional field. Models will be presented that are used to a priori understand the potential of a certain composition for structural applications. These models include the prediction of phases formed in the alloy, as well as the prediction of hardening and deformation mechanisms. Experimental results of the validation and comparison of these models will be presented, as well as how this type of methodology can lead us to deepen our fundamental knowledge of hardening and deformation mechanisms in concentrated metallic alloys. Great emphasis will be given to Cr-Co-Ni compositions, refractory alloys, and precipitation-hardenable alloys, three classesof alloys that have been extensively studied in the literature of HEAs. Ideas and paths forthe development of these alloys will also be discussed.

Highly porous CP-Ti foams manufactured with powder compaction, the space holder method and plasma-assisted sintering for biomedical applications

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Highly porous titanium foams are components used to replace bone structures with low elastic modulus due to their ability to avoid the phenomenon called stress shielding. The porous structure with a volume of over 70% reduces the elastic modulus of the component to values closer to that of trabecular bone, mimicking the mechanical characteristics of the structure being replaced. However, producing foams with porosities above 70% using space holders is challenging because the component can collapse during processing due to the high amount of temporary material. In this study, pure titanium powder was mixed with 50, 70, and 80 volume percent KCl as a space holder, cold-compacted, and sintered at 1250 °Cin a tubular furnace to obtain foams with high porosity. The space holder was removed inthe same furnace by sublimation prior to sintering. The porous architecture of the foams was analyzed through image analysis. The elastic modulus of the foams was measured using Ultrasonic Waves Transmission (UWT), a non-destructive method of mechanical characterization that is commonly applied to bulk materials and has great potential for exploring foams. The results showed an influence of the amount of space holder on pore , form and distribution. The elastic moduli measured in all samples with 80% porosity were equivalent to those of trabecular bone.

Hydrogen Storage Properties of the TiVFeZr Multicomponent Alloy

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Hydrogen as an energy vector is one of the possible solutions to the widespread use of renewable energy sources. Intermetallic-based multicomponent metal hydrides have been widely studied to storage hydrogen, such as TiZrCrMnFeNi[1], (ZrTiVFe)xAly[2] and ZrTiVAl1-xFex[3]. In this work, the structure and hydrogen storage properties of the TiVFeZr multicomponent alloy synthesized by arc melting was comprehensively investigated. The TiVFeZr alloy solidified forming a single C14-type Laves phase structure. This alloy presents fast hydrogen storage kinetics at room temperature with high hydrogen storage capacity, absorbing H/M = 1.5 (2.41 wt.%) in less than 100 s under initial hydrogen pressure of 20 bar. This is one of the highest hydrogen storage capacities among the Laves phase alloys reported to date. The formed hydride is highly stable, which demands temperatures above to 550 °C to completely desorb hydrogen. The high hydrogen storage capacity of this alloy as well as the high stability of the formed hydride are discussed in terms of the high fraction of hydride forming elements in the C14-type Laves phase structure.

Acknowledgments to the Serrapilheira Institute (process Serra-1709-17362), Sao Paulo State Research Support Foundation (processes 2017/16273-7, 2018/25154-4 and 2021/00912-6), the Brazilian National Council for Scientific and Technological Development - CNPq (Grant number: 309467/2021-7 and 407906/2022-3) and the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

References:

[1] P. Edalati, R. Floriano, A. Mohammadi, Y. Li, G. Zepon, H.W. Li, K. Edalati, Journal vol. 178, pages 387-390 (2020).

[2] X. Ma, X. Ding, R. Chen, W. Cao, Q. Song, Journal vol. 47, pages 8409-8418 (2022).

[3] X. Ma, X. Ding, R. Chen, X. Gao, Y. Su, H. Cui, Journal vol. 12, pages 11272-11281 (2022).

Individual effect of Y and Nd on the microstructure formation of Mg-Y-Nd alloys processed by severe plastic deformation

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The effect of sole yttrium and neodymium alloying on the microstructure formation during severe plastic deformation by equal channel angular pressing (ECAP) was investigated. The results are compared with a ternary Mg-4Y-3Nd alloy, which represents a simplified version of the commercially successful WE43 alloy. It is shown that the presence of Nd primarily caused precipitation during the processing of the Mg-3Nd alloy, while Y remained dissolvedin the magnesium matrix in the Mg-3Y alloy. This difference resulted in a significantly smaller average grain size in the Mg-3Y alloy (~0.77 nm) than in the Mg-3Nd alloy (~1.3μm) after the final step of the processing and formation of a slightly different texture. Consequently, the composition and the processing affected the mechanical and corrosionproperties of the investigated materials. It is shown that the ECAP-processed W3 sample exhibits a surprisingly good combination of ultrafine-grain structure, weak crystallographictexture, high strength, and high corrosion resistance compared with the other investigatedsamples. These attributes make this material very interesting for utilisation in the industry and/or medicine.

Influence of solution annealing temperature on the microstructure of maraging 400 steels

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Maraging 400 are a precipitation hardening steels whose base composition is Fe-13Ni-15Co-10Mo-1Ti (wt.%). In these steels, the annealing heat treatment needs to guarantee a solid solution and a martensitic microstructure, characteristics that provide the driving force for precipitation during aging [1-2]. For other classes of maraging steels (containing 18wt.% of nickel and lower contents of molybdenum and cobalt), the solution annealing treatment is very established (usually 820 °C for 1 h). However, the literature has indicated that for maraging 400 is required higher annealing temperature or time, due to changes in the chemical composition. [1-3]. The aim of this work was to study the influence of the annealing temperature on the microstructure of maraging 400 steels. The study were carried out on samples with five different compositions (varying molybdenum and titanium contents) solution annealed at 900, 1000 and 1200 °C for 1 h. Then, the samples were characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), electron backscatter diffraction (EBSD), dilatometry and Vickers hardness. The results indicated for an annealing temperature bellow 1000 °C, a microstructure containing undesirable Fe_3Mo micrometric precipitates. It was only at an solution annealing temperature of 1200 °C that it was possible to obtain a full martensitic microstructure. The molybdenum content influences the and amount of Fe_3Mo precipitates, as well as the phase transformation temperatures (of Fe_3Mo, austenite and martensite).

Acknowledgements: CNPq, Capes, Lawrence Berkeley National Laboratory - Molecular Foundry and Deutsches Elektronen-Synchrotron (DESY).

References:

[1] Schmidt, M.; Rohrbach, K. ASM International, p. 528-548, 1991.
[2] Fang, J. Y. C. et al. Frontiers in Materials, v. 9, p. 2-7, 2022.
[3] Niu, M. et al. Acta Materialia, v. 179, p. 296-307, 2019.

Mechanical properties and microstructure of biodegradable ultrafine-grained magnesium alloy processed by severe plastic deformation

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Biodegradable magnesium alloy Mg22Gd alloy was processed by high pressure torsion (HPT, up to 15 turns) at room temperature and the pressure of 2GPa. Mechanical properties were investigated by detail 2D microhardness measurement. HPT straining introduces a lateral inhomogeneity in the material and resulted in inhomogeneous distribution of microhardness throughout disk specimens with the minimum in central zones and increasing values towards the specimen periphery. The microhardness was found to increase with increasing strain imposed by HPT and tend to saturate at about HV=145. The microstructure (grain size, phase composition, phase morphology, etc.) evolution with strain due to HPT was investigated by light and scanning electron microscopy and EDS. High Gd content in the alloy resulted in the precipitation of stable Mg5Gd phase. During straining the phase was continuously fragmented and only tiny particles were found in heavily strained particles.Electron back scatter diffraction (EBSD) and automated crystallographic orientation mapping in transmission electron microscope (ACOM TEM) were employed to characterize the fragmentation of the initial coarse grain structure with increasing strain. HPT was found to result in strong grain fragmentation of the factor of approximately 1000.High density of dislocations of the order of 10^14/m2 was found in individual specimens. The dislocation density was determined by enhanced techniques of positron annihilation spectroscopy and X-ray diffraction.The strength of the material was correlated with the dislocation density. It was shown that hardening during HPT is caused mainly by dislocation strengthening due to interaction of glide dislocations with forest dislocation as a result of relatively homogeneous distribution of dislocations in deformed specimens. Unlike in other magnesium alloys processed by severe plastic deformation the strengthening by grain refinement is negligible in MgGd alloy.

Microstructural and physical properties of commercial Nb-46.5wt.%Ti superconducting wires with different configuration strategies

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Commercial superconducting wires of Nb-46.5wt%Ti alloys are used in a diverse range of applications related to medical, transportation, and high energy physics. The microstructure is composed by the Nb-rich superconducting phase together with α-Ti precipitated in a nanocrystalline structure, acting as pinning centers. The wires consist of a Nb-46.5%pTi multifilamentary structure embedded in copper and, depending on the configuration strategy, this might lead to distinct superconductivity behavior. The bundling and the processing route generate distinct morphologies and microstructures and, consequently, superconducting properties, primarily the critical current density. The objective of this work is to characterize three different commercial superconducting wires to observe the microstructure and correlate with the superconducting properties. The microstructure was characterized by Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). The SEM samples were prepared by mounting, grinding, and polishing; and the TEM samples by Focused-Ion Beam (FIB). The samples were characterized using a Physical Property Measurement System (PPMS) coupled with a Vibrating Sample Magnetometer (VSM), in which the characterizations were made to obtain the critical temperatures and the critical current densities at 4.2 K from loops of magnetization in moment vs. applied magnetic field curves.

Acknowledgments: This study was financed by the Brazilian National Council of Scientific and Technologic Development (CNPq; grant 408917/2022-9). This research used facilities of the Brazilian Nanotechnology National Laboratory (LNNano), part of the Brazilian Center for Research in Energy and Materials (CNPEM), a private non-profit organization under the supervision of the Brazilian Ministry for Science, Technology, and Innovations (MCTI). The SEM-FIB-FT staff is acknowledged for the assistance during the experiments (Proposal 20231986).

Multicomponent Fe-based glass-forming alloysdeveloped for coatings applications

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Fe-based glass-forming alloys are suitable to be used as coatings in steel components that require good corrosion and wear properties. We are using different strategies to design alloys with good compromise between these two properties, which are usually contradictories. In the present work we discuss the design strategies and the microstructural characterisation of multicomponent alloys of the following Fe-based systems: Fe-Cr-Ni-B, Fe-Cr-Mo-Nb-B, Fe-Co-Ni-Cr-Mo-B and Fe-Cr-Mo-Nb-B-C. The results show the important role and necessary balance played by the elements added to improve the glass forming ability, the ones added to improve the corrosion resistance and the ones that contribute to the formation of hard phases to improve the wear properties in partially crystallised alloys.

Nanostructured ceramic-metallic films produced by magnetron sputtering

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This study dealt with the deposition of nanostructured ceramic-metallic thin films produced by magnetron sputtering. Three groups (Ti-Si-O-N, Al-Ti-C and Al-Si-N) were deposited froma mixture of metallic and ceramic sputtering targets (Ti and SiO2, Al and TiC, and Al and Si3N4, respectively). Ti-Si-O-N films were deposited seeking an application on solar collector system Al-Ti-C group was designed aiming an application on mirrors for heliostats used on solar concentrating system and Al-Si-N films were deposited seeking the hardnening of Al films. Deposited films were analyzed by transmission electron microscopy to evaluate its morphology. X-ray photoelectron spectroscopy (XPS) and Rutherford backscattering spectrometry (RBS) were used to determine the films chemical composition. The nanohardness of Al-Ti-C and Al-Si-N groups was evaluated, while samples from Ti-Si-O-N group were optically analyzed on ultraviolet, visible and infrared radiation ranges. Analyses results show that films from every group are formed by metallic nanocrystals embedded in an amorphous matrix, where both the volumetric fraction of nanocrystals and films hardness are dependent on the amount of deposited metal. Films from Ti-Si-O-N group showed nanoparticles only on the high metal fraction layer, linked to the thermal emittance of sample the low metal fraction layer, related to solar absorption, did not show metallic nanoparticles.

Acknowledgements:

This work was funded by FAPERJ - Fundação Carlos Chagas Filho de Amparo à Pesquisa do Estadodo Rio de Janeiro - process SEI-260003/019684/2022.

On the impacts of reactive high-energy ball milling onthe structural, microstructural, and hydrogen storage performance of refractory multicomponent alloys

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Reactive high-energy ball milling (RBM) is a well-known technique for processing metal hydrides, often resulting in nanocrystalline materials with fast kinetics for hydrogen sorption. Thus, in this work, the influence of RBM processing on the structural properties ofa refractory alloy was investigated and correlated with hydrogen sorption properties. The TiVNbCr alloy was chosen since it has been extensively studied. The milled hydride was synthesized in two steps: (i) preparing the TiVNbCr alloy through arc melting; (ii) performing RBM under H_2pressure. The as-cast alloy crystallizes as a body-centered cubic (BCC) single-phase. Upon hydrogenation, the TiVNbCr alloy achieved a hydrogen-to-metal ratio (H/M) close to 2. Moreover, the XRD total scattering experiments followed by pair distribution function (PDF) analysis indicate that the hydrogenated alloy assumes a face-centered structure (FCC) typical for dihydride. Surprisingly, the X-ray experiments of the RBM hydride indicate a mixture of phases concerning a BCC monohydride and an FCC dihydride. The thermogravimetric analysis for desorption of the RBM powder indicates that the sample releases around 1.05 H/M. The comparison between the PDF analysis of the as-cast hydrogenated TiVNbCr alloy and the RBM hydride indicates that the reactive ball milling exerts a marked influence on the samples'' overall crystallinity, contributing to increasing the lattice defects and micro-strains. The accumulation of defects within the crystal structure can suppress the phase transformation from monohydride to dihydride, consequently diminishing the hydrogen uptake of BCC alloys [1]. Therefore, this work reveals the impact of ball milling on the structural characteristics of refractory high entropy alloys and proves its deleterious effect on their hydrogen storage performance.

[1] Q. Wang, X. Dai, C. Wu, Y. Mao, Y. Chen, X. Cao, Y. Yan, Y. Wang, H. Zhang, J. Alloys Compd. 830 (2020) 154675.

Reversible room temperature hydrogen storage in the $(Ti_{0.5}Zr_{0.5})_1(Fe_{0.33}Mn_{0.33}Cr_{0.33})_2$ multicomponent alloy designed by computational thermodynamic tools

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Multicomponent alloys with C14 Laves phase are promising hydrogen storage materials due to their ability to reversibly absorb substantial amounts of hydrogen at room temperature with fast kinetics and improved cyclability [1]. The applicability of these alloys as a hydrogen storage media is governed by their thermodynamic properties, which can be tuned by the chemical composition design. In this regard, design methods with predictive properties modeling are essential for exploring the vast compositional field of multicomponent alloys. In this work, a computational approach was used aiming at a C14 Laves phase alloy for room-temperature hydrogen storage. First, CALPHAD method was applied to predict the phase formation of the AB_2-type alloy compositions. Secondly, a model to predict the thermodynamics of the metal-hydrogen systems was proposed to calculate the pressure-composition-isotherm (PCI) of multicomponent C14 Laves phase alloys [2]. According to the design, the $(Ti_{0.5}Zr_{0.5})_1(Fe_{0.33}Mn_{0.33}Cr_{0.33})_2$alloy would solidify as a single C14 phase and would be capable of storing hydrogen reversibly due to the calculated hydrogen equilibrium pressure in the order of 10^1bar at 30 °C. The alloy was produced by arc-meltingand experimentally characterized by structural and volumetric techniques. X-ray diffraction analysis indicated the formation of a single C14 Laves phase. PCI diagram showed that the alloy reversibly stored 1.7 wt.% (H/M = 1) of hydrogen with fast absorption and desorption kinetics at 30 °C up to 100 bar. Furthermore, the alloy showed great cycling stability over50 cycles at 30 °C, exhibiting excellent reversible hydrogen storage properties.

Acknowledgments:

Serrapilheira Institute (grant number Serra-1709-17362), CNPq (grant number: 407906/2022-3), CAPES (Finance Code 001), Max-Planck-Institute.

References:

[1] F. Marques, M. Balcerzak. Energy Environ. Sci. 14, 5191-5227 (2021)

[2] J.B. Ponsoni, V. Aranda. Acta Materialia 240, 118317 (2022)

Seven decades of Hall-Petch: background and current trends

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The relationship between grain size and mechanical strength has been studied for many decades. An important step forward in this area was achieved seven decades ago with the publication of papers which pointed that the yield stress of mild steel is proportional to the inverse of the square root of the grain size. Similar trends have been reported for other metallic materials and this relationship has been accepted until the present days. The phenomenology behind this relationship is not clear yet though. Many mechanisms have been suggested to explain it but none of these mechanisms has been supported by a large amount of experimental data for different materials. There has also been disagreement regarding the thermal-activated nature of this effect and its validity for a broad range of grain sizes including nanocrystalline materials. The present study reviews the developments in this field of study and the recent advances which include large amount of data for materials with nanocrystalline structure and advanced characterization techniques. It is shown that the ability to produce samples with very small grain sizes, which was developed in recent decades, shed light in this topic and many trends are clearer now. It is also shown that a recently developed model display the best agreement with experimental data for approximately 30 different metals and alloys, tested at different strain rates and temperatures. The trends of grain refinement hardening at low temperatures and grain refinement softening at high temperature are both explained by this model.

Solubilization and aging heat treatments of aluminum alloy 5083 with Microstructural analysis via FEG-EBSD

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The evaluation of the compositional and microstructural characteristics of alloy 5083 subjected to solubilization and aging at different combinations of temperature and time, aims to develop thermal procedures in the recovery of this alloy that is susceptible to sensitization [1] in its application. Heat treatments were carried out on samples of alloy 5083 H112 sheet. Solubilization took place at 450ºC and 500ºC, with a soaking time of 2 hours and followed by cooling in water, and aging took place at 180ºC with soaking times of1, 3, 6 and 9 hours followed by air cooling. Then, the samples were evaluated via X-ray diffraction (XRD) and scanning electron microscopy (SEM-FEG with EDS and EBSD), in order to evaluate the most favorable microstructures for sensitization, by controlling the amount and of precipitates rich in Mg. It was observed in the original sample, partially work hardened, signs of incomplete recrystallization, a small amount of precipitates rich in Mg and grains of Al-α with a high content of magnesium in solid solution, indicating a metastable state, which prevailed in the samples. solubilized, as expected. In addition, the presence of coarse precipitates of Al6Fe was noticeable. Regarding the solubilized samples, they showed growth of thermally activated grains and incomplete recrystallization. The samples aged under different conditions, on the other hand, showed a greater amount of precipitates compared to the others, promoting a state closer to equilibrium as a function of temperature and soaking time.

Acknowledgements:

To the Human Resources Program of the National Agency of Petroleum, Natural Gas and Biofuels, for the financial support invested by qualified oil companies in the RD I clause of ANP Resolution nº 50/2015.

References:

[1] Y.K. Yang, T. Allen, Direct visualization of β phase causing intergranular forms of corrosion in Al-Mg alloys, Mater. Charact. 80 (2013) 76-85.

Strategies to design multicomponent metal alloys for different hydrogen storage applications

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Hydrogen has a central role in the fight against the climate change by being an excellent energy vector to store and transport renewable energy. Metal hydrides (MHs) are suitable candidates to store and transport hydrogen in solid state with high volumetric energy density. In the past few years, many attentions to multicomponent alloys that form multicomponent metal hydrides (MCMHs) have been given because of the possibility to tailor their hydrogen storage properties by compositional control. However, navigating the endless compositional field of multicomponent phase diagrams and finding the composition and phases with optimized properties is not a simple task. In this work, we present three alloy design strategies that were successfully employed to find MCMHs compositions with suitable properties for different applications. The first strategy was based on calculation of geometrical, electronic, and thermodynamic parameters that allowed us to design a BCC single phase Mg-containing MCMHs. The second and third strategy were based on thermodynamic calculation using CALPHAD method coupled with pressure-composition-temperature (PCT) diagram calculation using models developed by our research group. The second strategy was used to design BCC single phase alloys with tunable hydrogen absorption/desorption plateau pressure. Finally, the third method was employed to design single C14-type Laves phase intermetallics also with tunable hydrogen absorption/desorption equilibrium pressure. The three design strategies were experimentally verified by synthesizing the designed alloys with an appropriate method, followed by structural characterization and hydrogen storage properties measurements. Acknowledgement Fapesp process 2020/07707-6.

Study of the microestructural stability of the nitridedlayer of AISI H13 steel after temperature exposure

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Plasma nitriding is a thermochemical process of technological importance to reduce damage on the steel surface, improving die life. To the aluminum die casting die industry, the most important advantage is to improve its tribological characteristics, due the increase in surface hardness and the presence of residual compression stresses were able to slow down cracking, erosion / etching of aluminum and wear in the regions of the feed channels. However, the properties of this layer may change, due to the conditions of use of the die. The industrial environment submitted the nitrided surface to temperatures up to 600°C and, in the literature, there is not a clear understanding of the microstructural stability of nitrided surface properties. Therefore, the aim of this study is to evaluate the effect of temperature on the nitrided layer of AISI H13 steel, which is used by the industry for making dies and molds used in the hot forming or casting process of non-ferrous alloys. Samples were hardened and tempered to 45 HRC and then nitrided aiming for a layer with adepth up to 100 μm, without white layer. After nitriding, some samples were annelead in afurnace at 600°C for 30min, 2h, 5h, 10h and 100h. Some samples were tested formicrostructure, x-ray diffraction and residual stress. The nitrated layer, after being subjected to high temperature, presents a reduction of level in the residual tensions of compression, bringing about a process of tensions relief. The same effect is not so stronglyverified in the material hardness profile. Additional tests as chemical mapping of some elements through WDS techniques and characterization of nitrides particles high-resolution scanning electron microscopy, FEG, was important in this study to better understanding themicrostructure stability of the of the nitrated layers when exposed to higher temperatures.

The effect of Al, Cr and Mo additions in NbTiSiZrcoatings

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Since the early 1990s, Nb-Si intermetallic based alloys are seen as potential candidates for ultra-high temperature structural applications [1]. The development of innovative manufacturing techniques and the addition of multiple alloying elements to niobium-silicide alloys are considered mandatory to reach physical and chemical property goals [2]. This investigation takes an important step to process sound Niobium alloys containing high hardness low toughness silicide compounds, taking advantage of the in-situ synthesis of alloys during the processing of multilayers. The PTA-DED technique is explored to process Niobium alloy multilayers aiming to assess the impact of elemental powder mixture complexities on the in-situ synthesis, solidification paths, metallurgical stability to multiple thermal cycles of additive processing and the oxidation resistance. Progressive additions of 5at%Al, 5at%Cr and 2at%Mo were made to an elemental powder mixture with a composition of Nb24Ti18Si+1Zr (at%). Results show that the presence of Al and Cr, individually or together, induces more segregation during solidification, leading to the formation of anomalous and ternary eutectics. However, the addition of Mo to the powder mixture stabilizes the high temperature primary phases and mitigates the solidification of anomalous eutectic. Multiple thermal cycles lead to a decrease in thermal gradient, resulting in coarse microstructures with overgrown silicides. It is shown that oxidation performance is determined by the presence and extent of the solid solution in the microstructure.

Acknowledgements:

The authors would like to acknowledge CNPq and CAPES for the financial support.

References:

[1] M.G. Mendiratta, J.J. Lewandowski, D.M. Dimiduk, Metallurgical Transactions A 22 (1991) 1573-1583.

[2] Y. Li, X. Lin, Y. Hu, X. Gao, J. Yu, M. Qian, H. Dong, W. Huang, Corros Sci 173 (2020).

Thermodynamic and properties investigation of Cu modified niobium silicides diffusion coatings

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Niobium silicides coatings have been widely studied due to the combination of the silicides oxidation resistance and high-temperature creep resistance and relatively low density of niobium. To obtain more stable niobium silicide coatings with a double layer of silicides, NbSi2 and Nb5Si3, the literature presents the need for pack cementation processing at temperatures above 1200 ºC, increasing the cost of the process and making it difficult to control its variables. An alternative to this challenge would be to accelerate the synthesis of silicides by adding alloying elements that would enable the formation of the double layer of silicides at lower temperatures. In this research, the effect of adding Cu to the processing of niobium silicide coatings by pack cementation was investigated through thermodynamic and microstructural analysis. The processing was carried out with pack mixtures of Si with and without Cu at 1000 ºC for 6 hours. The HSC Chemistry software was used for thermodynamic analysis, and SEM, EDS, and nanohardness were used for coating analysis. Thermodynamic analysis shows that during processing, the formation of Si halides occurs mainly, and the Cu halides can be disregarded due to their low partial pressures. Deposition of only Si on Nb is confirmed by the formation of only Nb5Si3 and NbSi2 silicides in the coatings processed with pack mixture modified with Cu. Results show that coatings processed with only Si formed only the NbSi2 layer. In addition, coatings with Cu had thicknesses of 45.79 ± 2 μm, higher than coatings without Cu, which had 32.54 ± 2.38 μm. Coatings processed with Cu also showed a decrease in hardness (979 ± 65.33 HV) compared to those without Cu (1110.4 ± 45.34 HV). The results indicate that although Si and Cu do not co-deposit on Nb, the element impacts the formation and properties of silicide coatings, accelerating synthesis and possibly inducing more defects in the silicide structure, resulting in a decrease in their hardness.

The voids on high coercive Sm-Co films deposited on Ta buffer

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Besides the wide discussion of Ta as a buffer layer in the literature, an investigation into the influence of the transformation from β-Ta to α-Ta phases in heat-treated Sm-Co films remains unexplored. Here, the use of the Ta buffer and the capping layer on the Sm-Co thinfilms leads to an optimization of the magnetic properties. The hysteresis shows that, in thiscase, the Sm-Co films present high coercivity values, greater than 4 T, and a quadrature factor approximately equal to 1. The observed improvement in the performance of the Sm-Co films seems to be related to the concomitance between the crystallization process of theSm-Co films and the Ta transformation that occur during annealing. In particular, X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) results indicate theoccurrence of an intense diffusion process between layers, leading to the formation of thecrystallized $TaCo_3$phase. Additionally, transmission electron microscopy (TEM) analyzes show the presence of voids in the microstructure of the Sm-Co layer, which seem to act aspinning, providing the high values of coercivity observed. Acknowledgments: This work wasfunded by the Brazilian research agency CAPES.

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Advances in the investigation of kinetic parameters ofthe Al-V system

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Vanadium alloys are investigated as cladding materials for nuclear fusion reactors, due to their low activation and good mechanical properties over a wide range of temperatures. However, the main problem of these alloys is that they exhibit a poor oxidation resistance at high temperatures [1], so they need external protection, as aluminide coatings. Also, it is vital to know the kinetic data of phases of the Al-V system, with the purpose of estimating the thermal stability of substrate/coating systems. The present work aims to determine kinetic parameters (growth constants and interdiffusion coefficients) in Al-V diffusion couples using an unconventional method, namely the pack cementation technique. Samples were cut from a pure V plate and arc-melted 66V-44Al (at. %) ingots. The aluminide coatings were produced using the halide-activated pack cementation (HAPC) technique. Assuming that diffusion in the gas phase is not a rate-limiting step, the coating formation can be understood as a classical semi-infinite diffusion couple. The samples were processed by the HAPC technique from 800 to 1000°C, for 4, 9, 16 and 25 hours. The coated substrates were microstructurally characterized by XRD, SEM and EDS. The solid state diffusion governs the growth of the coating layers, given that the thickness variations follow a parabolic law. The growth constants were obtained by linearizing the kinetic curves for each phase and they were used to calculate the interdiffusion coefficients, with the Wagner' s approach (1969) [2]. The kinetic parameters were calculated for the VAl_3, V_5Al_8 and (V) phases. The VAl_3 phase has interdiffusion coefficients approximately one to two orders of magnitude higher than the those of V_5Al_8 and (V).

Acknowledgements:

The authors acknowledge the financial support from FAPESP (grant #2022/06285-6) and CAPES (Finance Code 001).

References:

[1] Muroga, T., Compr. Nucl. Mater., v.6, 1-18, (2020).
[2] Wagner, C., Acta metallurgica, v.17 n.2, 99-107, (1969).

Analysis of heat threated aluminum alloys 6351subjected to equal channel angular pressing

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The production of aluminum and its alloys is one of the most used in the metallurgical industry and with increasing use in aeronautics, automotive and shipbuilding. Because pure aluminum has little mechanical strength, other elements are added in alloy forms to meet the engineering applications, as is the case of alloy 6351, according to this understanding, for this work the alloy was subjected to thermal treatments, for the formation of a dense and fine dispersion of particles within the alloy to promote increased mechanical strength, and also a process of severe plastic deformation called ECAP (equal channel angular pressing) aiming to maximize the refinement in grain to promote the improvement of mechanical strength [1]. In this research the effect of ECAP on alloy 6351 samples subjected to heat treatments of solubilization, full annealing, and solubilization followed by aging will be analyzed. The process follows the introduction of each treated specimen into the die that consists of two channels of equal cross section that cross at an angle of 110°, and is extruded with the help of a punch. The processing temperature (150°C) and pressing speed (10mm/min) will be standardized [2], for later comparison, and the processing route (Bc) will be added. The microstructural analysis will be evaluated by optical microscopy (OM) and X-ray diffraction (XRD), and the mechanical properties by Vickers microhardness.

References:

[1] THIYAGARAJAN, R. ; GOPINATH, A. Enhancement of Mechanical Properties of AA 6351 Using Equal Channel Angular Extrusion (ECAE), Materials Science and Metallurgy Engineering, Vol. 2, pp. 26-30, (2014).

[2] COSTA, F. Study of the Behavior of Aluminum Alloys 6351 t6 and 7075 t651 Submitted to the Extrusion Process in Angular Channel, Thesis - Post-Graduation in Materials Engineering at the Federal Institute of Piauí (IFPI), 102 p. (2022).

Analysis of the effect of simulated corrosion on the mechanical properties of biodegradable magnesiumalloy WE43

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Magnesium and its alloys are being considered to replace bioinert metallic materials in the fabrication of temporary trauma implants because they are biodegradable in the human body, making a second surgery for their removal unnecessary. However, a barrier to their implementation in orthopedic applications is the lack of knowledge about the effects of biodegradation on the structural integrity of this material. In this study, a numerical comparison of the mechanical properties of the WE43 magnesium alloy is carried out in order to predict the effects of localized and stress corrosion on its structural integrity and to differentiate the influence of this phenomenon. Therefore, the mechanical behavior of the alloy was simulated by means of a von Mises elastoplastic model, coupled to a continuous damage model of localized pitting corrosion and stress corrosion cracking, through the development of a subroutine (VUMAT) in the Abaqus®/Explicit software. Subsequently, this corrosion model is calibrated according to the data obtained from mechanical characterization and in vitro degradation tests. According to results, it is possible to note that both localized and stress corrosion models are able to capture the effects of reducing the fracture loss mass with increasing mechanical loading. Regarding the influence of stress corrosion on the mechanical properties of the material, the small difference in the mass loss at fracture value found between the two corrosion models suggests that the localized corrosion mechanism is the main influencing factor in reducing the structural integrity of the material caused by corrosion.

Analysis of the effects of normalization on gray castiron brake disc samples

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Gray cast iron is a ternary alloy of Fe-C-Si with percentages above 2% carbon, its properties, such as high degree of hardness, wear resistance, vibration damping, indispensable given wide use, being one of the metals consumed in large scale for production of products, mainly in the secondary sector (metal manufacturing), especially for the automotive industry. The objective of this work is to perform comparative analysis of the microstructure of the part and the hardness obtained aiming to find arguments to corroborate the claim that the sample of a standard brake disc has been manufactured from gray cast iron. The comparison method of visual analysis of the samples will be used (raw and treated) via images from researched literature, as well as explaining the steps, from cooling in still air outside the furnace (muffle type), followed by grinding (sandpaper from 80 to 1500), polishing (alumina of 0. 05, 0.3 and 1 micrometer alumina), chemical attack with 3% nital reagent (with time around 15 seconds of attack), and even obtaining data for the Rockwell C hardness test. In summary, it is concluded that, normalization is not usual in the manufacture of brake discs, due to the reduction of hardness, which is primordial to the mechanical property of wear resistance.

Analysis of the metallurgical effects of bismuth addition on the machinability of the alloy Al-12.6wt.% Si: temperature and cutting tool wear criteria

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Al-Si alloys are widely used in industry to manufacture components that are exposed to critical wear conditions, as they provide better mechanical properties and higher resistance to corrosion and defect propagation [1]. Machining is one of the most widely used manufacturing processes in industries, however Al-Si alloys are characteristic to present low machinability, especially those with more than 12.0wt% of solute content, because of the great tendency of these alloys in adhering on the cutting tools [2]. This is a motivation factor to the development of research that investigate the effect of alloying elements, such as bismuth, to improve the machinability of the Al-Si system and that can predict microstructural conditions and solidification parameters that facilitate the machinability of these materials. Thus, an experimental study was conducted with Al-12.6wt.%Si and Al-12.6wt.%Si-3.2wt.%Bi alloys solidified in a water-cooled vertical directional device to establish correlations between microstructure and average cutting temperature during necking processes on a bench lathe. From the experimental results, a power-type function was obtained, which relates the cutting temperature according to the eutectic spacings, from the base to the top of the ingot, and was observed that the cutting temperature decreased with increasing eutectic spacings. Besides, the results show that the addition of 3.2wt.% of bismuth in the Al-12.6wt.%Si alloy increased the cutting temperature, however decreased the wear of the cutting tool, which remained constant for the samples analyzed.

Acknowledgements:

The authors are thankful for the structure and financial support provided by IFPA, UFPA, FAPESPA, CAPES, and CNPq for the development of this work.

References:

[1] D.B, Carvalho et al., Materials Research, vol. 16, 874-883 (2013)

[2] R.V, Reyes et al., Philosophical Magazine Letters, vol .96, 1-10 (2016)

Analysis of waste of metallic structural materials incivil construction

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The present work consists in a case of study focused on the analysis of civil construction waste, resulting from the construction of a single-family residence in the city of Teresina-PI. The study begins with a bibliographic review, where multiple concepts about civil construction waste are presented, aiming to improve the understanding of the term for a better experience in this work. Subsequently, methodologies are presented not only to classify the waste found, but also to prevent, mitigate and combat all types of losses. The analysis of the residential waste object of this paper starts from the earthworks until shortly before the stage of completion, passing through the planning, foundation, installations, roofing and another phases. The research showed that, even with the advancement of technologies to combat civil construction waste, a more rigorous control and a better awareness of the need to implement practices aimed at reducing losses in works in search of improvements for the environment, the company and the consumer, are still required, and the practices to combat waste presented in this work are an excellent way to initiate this change.

Analytical techniques to study the contamination byheavy metals on the beaches of Sepetiba Bay

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The Sepetiba Bay occupies an area with great natural resources and high viability for undertakings related to the processing and transport of ores. Given this scenario, since 1950, investments in the mining sector have been increasing. Nowadays, there are more than 100 companies in the sector that generate ores and residual metals, leading to devastating environmental consequences in the hydrographic basin, such as the reduction of the Atlantic Forest, water contamination, severe reduction of fishing area, contamination of fauna and flora [1]. In this context and with the objective of evaluating the current advance of contamination in the region, the present research analyzed the content of metals in the sands of the beaches belonging to the three cities that border the Bay: Itaguaí, Mangaratibaand Rio de Janeiro. Through a factorial design of experiments, 9 beaches and two sources of contamination were selected. Two quantitative factors were evaluated: distances between the beach and each source of contamination and a qualitative factor (season: summer or winter). The response variable was the metal content determined using the x-ray diffraction (XRD) and x-ray fluorescence (FRX). The ANOVA and the response surface methodology were used to statistically evaluate the degree of soil contamination. A second-order regression model showed the statistical significance of contamination focus 2 and the influence of the season. This last result corroborates published studies on the low rate of water renewal during the summer months [2]. The results obtained allowed identifying the degree of contamination increase to establish a possible recovery plan.

Acknowledgements:

FAPERJ

References:

[1] N. L. C. O. Tapanes, R. Gaidzinski, G. S. X. Marti G. S. B. Mendes, T. C. Santos, R. V. Alcantara, Contaminação de resíduos metálicos na Baía de Sepetiba. Vídeo (2022)

[2] A.L.F. dos Santos, R.S. Peixoto, P.A. Rosman, P.C.C. Rosman, www.sisbahia.coppe.ufrj.br (2018)

An evaluation of stress corrosion cracking sensitivity in heterostructured AA1050/AA7050 Al sheets

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In recent years, heterogeneous materials have been emerged as a new way to optimize properties taking advantage of mechanical behavior of two or more alloys in one bulk sheet [1]. The Accumulative Roll- Bonding (ARB) is a suitable process for heterostructured sheets production, since it achieves mechanical bonding between dissimilar metals or alloys [2]. In this work, heterostructured sheets were produced using two Al alloys: the age-hardenable AA7050 alloy and the highly ductile AA1050 alloy. The former has been extensively investigated in order to achieve a good combination between high specific strength and corrosion resistance [3]. However, the existing research is insufficient to stablish a correlation between heterostructures and corrosion properties, and thus stress corrosion cracking (SCC) sensitivity of these materials has far less been investigated. Firstly, sheets of both alloys in the annealed condition were stacked and then rolled with a thickness reduction of 50% in a single pass at 450 °C or 500 °C. The roll-bonded sheets were wire-brushed, cleaned, cut and stacked, pre-heated for 5 minutes at the process temperature and the procedure was repeated up to eight times. Therefore, the effect of processing parameters (number of cycles and temperature) on the SCC sensitivity using slow strain rate (SSR) tensile tests in a 3.5 wt.% NaCl aqueous solution were examined. Microstructure evolution, tensile behavior and fracture surface analysis were discussed to address the combined effects on the SCC sensitivity.

Acknowledgments: The authors thanks to CNPq (Grant numbers 153585/2018-8 and 160274/2019-2) and FAPESP (Grant number 2016/10997-0).

References:

[1] Y. Estrin, Y. Beygelzimer and R. Kulagin, Adv. Eng. Mater. 21, 1900487 (2019)
[2] H. Yu, et al., Sci. Rep. 3, 2373 (2013)
[3] D. Yuan, et al., Mater. Sci. Technol. 37, 616 (2021)

Application of different Ti cathodic cage plasma deposition treatments: Influence on mechanical and tribological properties of AISI 304 stainless steel

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AISI 304 steel is an austenitic stainless steel used in various applications, such as equipment for chemical, petrochemical, and food industries, biomedical prostheses, mechanical construction, among others [1,2]. Thin films were deposited on AISI 304 stainless steel substrates using different plasma-based PVD (Physical Vapor Deposition) techniques, namely duplex, simultaneous duplex, and deposition, using a Ti cathodic cage. The objective was to evaluate the influence of the applied technique on the mechanical and tribological properties of the films. The samples were analyzed using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) characterization techniques, as well as Vickers microhardness, Rockwell C adhesion and wear resistance tests. The results showed an increase in surface microhardness for all films obtained, with good adhesion characteristics, especially for the samples from the duplex and simultaneous duplex treatments. The simultaneous duplex treatment sample exhibited the best wear resistance results when compared to the other treated samples and the untreated substrate, highlighting the efficiency of this technique in obtaining films with good mechanical and tribological properties obtained in shorter treatment times than conventional duplex treatment.

Acknowledgements:
Conselho Nacional de Desenvolvimento Científico e Tecnológico - CNPq, Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - CAPES, Fundação de Amparo à Pesquisa doEstado de Alagoas - FAPEAL, Laboratório de Plasma - LABPLASMA (UFPI).

References:
[1] CISQUINI, P. et al. Effect of the roughness produced by plasma nitrocarburizing on corrosion resistance of AISI 304 austenitic stainless steel. Journal of Materials Research and Technology, v. 8, n. 2, p. 1897-1906, abr. 2019.
[2] SILVA, A.; MEI, P. Aços e ligas especiais. 3a ed. São Paulo: Blucher, 2010.

Arrhenius coefficients for the oxidation reaction of mechanical construction steel

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From both a metallurgical and economic stand point, it is extremely interesting to understand the influence of temperature on the oxidation rate of distinct steel alloys, since, depending on composition and temperature different oxidation mechanism can be identify [1]. This is specifically important during hot working of steel where the oxidation products are undesirable and their formation should be minimized to reduce production cost [2]. In this work two mechanical construction steel were studied at temperatures ranging from 900°C to 1200°C. The kinetic parameters were obtained from high temperature oxidation tests carried out in an unsealed PID-controlled oven for 0,5 h to 4 h. The samples masses were measured prior and after the experiment and the difference was used to calculate the overall gain of mass. The results have shown that the kinetics parameters of the AISI 1045 and 4340 alloys, are very similar, 104.449 J.mol-1, and 98.937 J.mol-1, respectively, both following a parabolic oxidation mechanism. Nevertheless, since the oxidation rates of both alloys were lower than that that of pure iron [3], carbon content and other alloying elements may have hindered oxides formation. Besides, the SEM/EDS analyses revealed a very porous oxide layer containing a carbon gradient decreasing from the metal-oxide to the oxide-air interfaces, suggesting carbon diffusion from the metal to the oxide, followed by its oxidation to CO2 in the presence of atmospheric O2.

Acknowledgements:
The authors would like to acknowledge the FCMF foundation.

References:
[1] BIRKS, N.; MEIER, G.H.; PETTIT, F.S. High Temperature Oxidation of Metals. 2°Ed. Cambridge University Press, 2006.
[2] WU, H. et al. Oxidation Behaviour of Steel During hot Rolling by Using TiO2-Containing Water-Based Nanolubricant. Oxidation of Metals, p.315-335, 2019.
[3] CHEN, R.Y.; YUEN, W.Y.D. Review of the high-temperature oxidation of iron and carbon steels in air or oxygen. Oxidation of Metals, v. 59, p. 433-468, 2003.

Assessment of the apparent atomic volume by first principle calculations

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Measuring the volume of an atom is a complex task due to its dependence on multiple factors, such as the number of electrons in the atom, the type of bond it forms, and its nearest neighbors, etc. As a result, accurately measuring the atomic volume is not always possible. In this work, we present a functional methodology for measuring the Apparent Atomic Volume (AAV), which is the volume occupied by the atoms in a unit cell. To determine the AAV, we utilized Density Functional Theory (DFT) calculations and X-ray diffraction analysis with a synchrotron source. We examined pure Ni, as well as single-phase FCC systems of NiPd, NiV, CrCoNi, CrConiV, and CrCoNiPd alloys. The DFT results demonstrated a strong correlation with our XRD findings and were consistent with previously published experimental data. Moreover, the methodology enables to obtain functional measurements that can be used to correlate with the mechanical properties of metals and their alloys. This approach can be a valuable tool in enhancing the intelligent design of alloys and more precise control over their properties. Acknowledgements: The authors would like to express their gratitude to Fundação de Amparo à Pesquisa do Estadode São Paulo (FAPESP - grant 2021/12589-5)References: [1] W. Woo, Y.S. Kim, H.B. Chae, S.Y. Lee, J.S. Jeong, C.M. Lee, J.W. Won, Y.S. Na, T. Kawasaki, S. Harjo, K. An, Competitive strengthening between dislocation slip and twinning in cast-wrought and additively manufactured CrCoNi medium entropy alloys, Acta Materialia, 246 (2023) 118699.[2] Y. Mishima, S. Ochiai, N. Hamao, M. Yodogawa, T. Suzuki, Solid Solution Hardening of Nickel mdash;Role of Transition Metal and B-subgroup Solutes mdash, Transactions of the Japan Institute of Metals, 27 (1986) 656-664.[3] S. Nath, S. Bhattacharya, P.K. Chattaraj, Density functional calculation of a characteristic atomic radius, Journal of Molecular Structure: THEOCHEM, 331 (1995) 267-279.

Automatic System for Control, Acquisition and Storageof Electrical Resistance Measurements of Materials Depending on Temperature

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The search for new materials involves a great effort in evaluating their properties. One such property of interest is the electrical property. This work presents the SAME, an automatic electrical measurement system, whose operating principle is the Acquisition, Storage, and Treatment of Electrical Resistance Measurements of materials versus temperature variation. The electrical characterization involves knowing the behavior of its electrical resistance related to temperature. For thin films, this temperature range of interest extends from room temperature to about 700°C in several cycles, which imposes a very high dedication time by researchers. The system produced in the thin film laboratory at LAMAV/UENF integrates a temperature control system of a rudimentary oven, a bench multimeter for acquiring resistance measurements and a microcomputer connected to the multimeter to record and process the data obtained. All man-machine interaction is done from the computer in a program written in Python, the graphical SAME, which presents an interface where the operator must configure the temperature parameters, number of cycles and desired time for stabilization. A second application, the statistical SAME, performs the statistical treatment of the measurements obtained and issues the report with graphs representing the behavior of the electrical resistance of the material versus temperature. The average time to carry out the electrical characterization of a sample was 1415 minutes of manual availability by the researcher and, with the implementation of SAME, this time was reduced to 5 minutes, which is the average time spent setting up the experiment. Thus, the SAME was satisfactory in terms of the results obtained in relation to the repeatability of the tests and the convenience of the users.

Acknowledgments:
IFF, UENF, Capes

CHARACTERIZATION OF A 2205 DUPLEX STEEL MODIFIED WITH BORON OBTAINED VIA POWDER METALLURGY

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Stainless steels are commonly used when a good balance between mechanical properties and corrosion resistance is required [1]. However, depending on application conditions, they may have low wear resistance. Usually, to increase wear resistance, hard particles are added (carbides, borides, nitrides and others.) to the more ductile matrix (ferrite or austenite). As the addition of boron leads to the formation of primary and eutectic borides,which harden the material as solidified. [2] Traditional techniques such as casting and machining are difficult to apply. Therefore, this work will investigate the viability to preparea modified 2205 duplex steel with 2.5% wt. of boron through powder metallurgy. This technique is an alternative way to process materials that are normally difficult to form bytraditional methods. Consequently, this study involves investigate the influence of temperature on sintering process, as well as the study of microstructure formation, thermaland mechanical properties. The samples were prepared from a pre-alloyed metal powder bycold uniaxial compaction and sintered in a controlled atmosphere.

Acknowledgements:

The author is grateful for the funding of this study provided by CAPES (Higher Education Personnel Improvement Coordination - Brazil) and for the Professor Doctor Juliano Soyama for all support and orientation in this research.

References:

[1] G. Chail and P. Kangas, “Super and hyper duplex stainless steels: Structures, properties and applications,” Procedia Struct. Integr., vol. 2, pp. 1755-1762, 2016, doi: 10.1016/j.prostr.2016.06.221.

[2] M. A. Doñu Ruiz, N. López Perrusquia, G. Urriolagoitia Sosa, L. H. Hernández Gómez,and D. Sánchez Huerta, “Mechanical behavior on Cr base steels surface treatment,” DefectDiffus. Forum, vol. 365, pp. 148-153, 2015, doi: 10.4028/www.scientific.net/DDF.365.148.

Characterization of Metal Matrix Composites (MMC) ofTiC in Ni-base alloy matrix produced by laser alloying process

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Ni-based alloys are used for a wide variety of applications, with the majority of them being involved in corrosion resistance and/or heat resistance. These applications include aircraft turbines, heat-treating equipment, and the chemical and petrochemical industries. The addition of high-hardness carbides to these alloys can increase their wear resistance, thus expanding their range of use. In this study, mixtures of powdered nickel alloy (used as the matrix bonding material) and titanium carbides (used as the hardening ceramic phase) were utilized to produce metal-matrix composites (MMC) layers with the aim of achieving superior tribological performance. Nickel-based powder, combined with 20%, 40%, and 60% TiC agglomerated with an organic binder, was deposited as a slurry onto samples of AISI 1020 steel. Subsequently, the coatings were melted using the laser alloying process. The layers and substrate were characterized through optical microscopy, Vickers microhardness, and micro-adhesive wear tests. All coatings outperformed the substrate in terms of hardness and wear resistance. TiC additions above 40% led to the agglomeration of carbides.

Acknowledgements:
The authors acknowledge the financial support of São PauloResearch Foundation (FAPESP) - grant numbers: 2016/11309-0 and 2019/26362-2.

References:
[1] L. Zhu et al., Recent Research and Development Status of Laser Cladding:A Review, Opt. Laser Technol. vol. 138, 106915 (2021)
[2] A.A. Siddiqui and A.K. Dubey, Recent Trends in Laser Cladding and Surface Alloying, Opt.Laser Technol. vol. 134, 106619 (2021)

Characterization of the parameters of the stress-strain curve in high nitrogen austenitic stainless steels

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High-nitrogen austenitic stainless steels have been used in the manufacturing of orthopedic implants subjected to severe loads over long periods of time due to their excellent mechanical properties, good biocompatibility, and affordable cost compared to titanium alloys. However, research indicates that the percentages of Ni, N, and Nb have been harmful to the human body and pose challenges to the production and processing of the alloy, resulting in structural, microstructural, and mechanical defects. [1,2] Physical simulation of thermomechanical processing allows for estimating the dependence of thermomechanical processing parameters on the mechanical-microstructural responses of the alloy under conditions similar to industrial settings.[3] In this research, the competition between dynamic recovery and dynamic recrystallization is characterized through constitutive analysis of stress-strain curves obtained from continuous isothermal hot torsion tests at different temperatures (900-1200 °C) and deformation rates (0.01-10s^{-1}), along withan estimation of dislocation density along the plastic flow curve. The results show that the activation energy for hot deformation (Q_{def} = 594 kJ/mol) is high compared to the class of austenitic steels, delaying the mechanisms of dynamic softening (DRV-DRX) and being significantly affected by the moderate value of the stacking fault energy ($\gamma_{SFE} \sim 68.7$ mJ/m^2) and fine precipitates of the Z phase (CrNbN) present at grain boundaries, which shape the stress-strain curves.

Acknowledgements:

Fundação de Amparo à Pesquisa e ao Desenvolvimento Científico e Tecnológico do Maranhão

References:

[1] YANG, Ke; REN, Yibin. Science and technology of advanced materials (2010).

[2] YANG, Yixun et al. Acta Metallurgica Sinica (English Letters), v. 33, p. 307-312 (2020).

[3] POLIAK, E. I; JONAS, J. J. Acta materialia, v. 44, n. 1, p. 127-136 (1996).

Characterization of Waste from Sustainable Hydrogen Generation via Metal-based Pathways

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The high global demand for energy has led to the search for a sustainable energy matrix. The hydrogen-based economy has emerged as a possible solution to the energy problem, as hydrogen is the known fuel with the highest calorific value in mass basis and is the simplest and most abundant gaseous element in the universe [1]. Its generation can be obtained by metallic ways, e.g. the oxidation of aluminum in alkaline solution, hereupon the use of recyclable metals is an option for sustainable hydrogen production processes [2]. However, as in any chemical reaction, the generation of hydrogen can result in wastes, being crucial to characterize them in order to understand their properties, evaluate their reuse and proper disposal. Thus, the waste generated was subjected to three characterization techniques: Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Thermogravimetric Analysis (TGA). SEM provides high-resolution images of the morphology and structure of the surface of the waste, enabling detailed information to be obtained about shape, size, particle distribution and surface roughness. AFM analyzes topography and surface mechanical properties at the nanometer scale, including 3D measurements and information on hardness, elasticity and adhesion. While the TGA allows analyzing the thermal behavior of the waste, including changes in mass, presence of water, decomposition of compounds, thermal stability of the material and determination of purity. Thereby, looking at sustainable hydrogen generation as a promising prospect for technological advancement and energy migration, it is essential to fully understand the hydrogen generation reaction and characterize the obtained product in order to establish a sustainable process chain.

Acknowledgments:
To UFPR, CAPES, and the advisors professors.

References:
[1] ZÜTTEL, A. Naturwissenschaften, v. 91, p. 157-172, (2004).
[2] WANG, H. Z. et al. Renewable and Sustainable Energy Reviews, v. 13, p. 845-853, (2009).

COPPER NANOWIRES COATED WITH PVP FOR USE IN NANOFLUIDS

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The economic development of nations is related to how they use their sources of energy. In 2016, the consumption of energy in the industrial refrigeration sector was 51% of final energy use1. Traditional refrigeration systems use refrigerators such as water, air, hydrocarbons, ethylene glycol, etc. A viable alternative that aims to improve the properties of fluids is adding nanowires (NW) dispersion in those fluids, getting a colloidal sol called nanofluid (NF)2. CuNW's were chosen because of the lower cost of the metal, the abundance of it and the good conductivity when compared with silver nanowires3. The present work aims to obtain NWs produced from the synthesis and modification of copper functionalized with polyvinylpyrrolidone using an electric pressure cooker. These materials were obtained by the chemical reduction method. The obtained samples were characterized by XRD to identify the crystalline phases and FTIR spectroscopy to assign chemical composition to the samples. The formation of the CuNW material is dependent of the type and time of stirring, which were possible to obtain with 12 hours of vigorous stirring. With the FTIR spectra, it was possible to identify PVP coating the NW's, however with some features of oleilamine (OE) and oleic acid (OA) in the surface. Future studies are necessary to understand the effects of OE and OA in the CuNW materials.

Correlation between interfacial heat transfer coefficient, primary dendritic arm spacings, andmicrohardness in the horizontally solidified Al-11wt.%Si alloy

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It is important to know the variation of dendrite arm spacings during the solidification process to analyze microsegregation pattern which influences the homogenization kinetics, ultimate tensile strength, ductility, toughness and yield tensile strength of solidified alloys [1]-[3]. The main purpose of this work is to present a theoretical-experimental study for the prediction of the interfacial heat transfer coefficient during the horizontal directional solidification of an Al-11wt.%Si alloy on water-cooled stainless-steel chill under transient heat flow conditions. Six thermocouples were connected with the casting and the time-temperature data were recorded automatically. A numerical technique which compares theoretical and experimental thermal profiles was used to measure the heat transfer coefficient values. This has permitted the evaluation of the variation of this thermal parameter along the solidification which is represented by a power equation that shows the time dependence during the process given by h_i=constant$(t)^{(-n)}$, representing the best fit between the experimental and calculated curves. The obtained results also include the variation of primary dendritic arm spacings of alloy analyzed as a function of interfacial heat transfer coefficient. These dendrite arm spacings were found to decrease as the values of this coefficient are increased. Finally, an experimental law of the Hall-Petch type is proposed relating the resulting microhardness to the heat transfer coefficient investigated.

[1] M. Tiryakioğlu, Metall Mater Trans A Phys Metall Mater Sci, vol. 50, no. 7, pp. 3030-3032, Jul. 2019, doi: 10.1007/s11661-019-05257-2.

[2] J. Wang et al., Opt Laser Technol, vol. 147, p. 107606, Mar. 2022, doi: 10.1016/j.optlastec.2021.107606.

[3] I. Kaldre, A. Bojarevičs, M. Milgrāvis, T. Beinerts, and M. Kalvāns, Materials Research, vol. 26, 2023, doi: 10.1590/1980-5373-mr-2022-0330.

Correlations of mechanical properties by SPT (Small Punch Test) and conventional tensile test for Al 6061 –T6

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The Small Punch Test (SPT) was development by nuclear industries to analyses mechanical properties of irradiated materials principally by small volume of the samples. This technique intend to evaluate the materials behavior during the time life of nuclear reactors, where yours properties changed by irradiation intensity and exposition time. It is considered an almost ¨non-destructive” method [2] due to small sample volume and its applications are spreading for use in situations where conventional methods do not apply. SPT consists of pressing a sphere, with a diameter equal to 2.5 mm, in a miniaturized sample of circular geometry (diameter d = 8 mm and thickness about 0.5 mm)[1], which has fixed edges, tested in conventional mechanical testing machines with the aid of a device developed for their achievement. In this work, mechanical properties of aluminum (Al 6061-T6) were abstained by two different methods: conventional tensile test and the small punch test (SPT). The SPT results depends on graph interpretations and discussions take place at now. Correlations of results guide us in choosing the most appropriated method for interpreting the force x displacement graph from SPT.Acknowledgements: We thank Dr. Arnaldo H. P. Andrade for his invaluable collaboration (in memoriam)References:[1] ASTM E3205-20 Standard Test Method for Small Punch Testing of Metallic Materials, Annual Book of ASTM Standards, Part 03.01, ASTM International, 2020[2] M. F. Moreno, Effects of thickness specimen on the evaluation of relationship between tensile properties and small punch testing parameters in metallic materials, Materials and Design, 157 (2018) 512-522.

Corrosion resistance of AA2024-T3 anodized in a molybdate containing TSA bath and post-treated in aCe containing solution

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Aluminum Alloy (AA) 2024-T3 is a high-strength heat-treatable alloy used in aircraft design as structural and skinning material for the fuselage, due to the combination of good mechanical properties and light weight. The thermomechanical processing of the alloy increases its mechanical resistance; however, it also generates a complex and heterogeneous microstructure susceptible to localized corrosion. Traditionally, protection of aerospace AA is performed in Cr(VI) containing anodizing and post-treatment solutions. However, environmental concerns and stricter legislation on the use of such chemicals require the development of low-cost and environmentally friendly processes. As an alternative, tartaric-sulfuric acid (TSA) anodizing has been proposed. It is reported that the addition of corrosion inhibitors to the anodizing bath and/or their use in a post-treatment step can improve the anticorrosive properties of the anodic layer. We investigated the effects of adding molybdate ions to the TSA bath followed by post-treatment in a Ce containing solution on the corrosion resistance of AA2024-T3. The investigation was carried out by combining characterization (RBS, TEM and SEM-EDS) with electrochemical (EIS) techniques. RBS and TEM indicated the incorporation of Mo and Ce ions in the anodized layer; while the SEM analysis showed that a thicker oxide layer was obtained for the samples anodized in the Mo-TSA bath when compared to the TSA bath and that the post-treatment with Ce reduces the thickness of the layer. The EIS analysis in 0.1 mol.L-1 NaCl revealed improved corrosion resistance for the samples with Mo and Ce io while fitting of the EIS diagrams with electrical equivalent circuits showed that the inhibitors improved the protective properties of the oxide layer without modifying the protection mechanism.

Acknowledgements:
FAPESP Proc. 2018/01096-5 and CNPq Proc. 400895/2014-5

Design and characterization of lightweight steels reinforced by TiB_2

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The lightweight steels of the FeMnAlC system were developed due to the automotive industry''s demand for materials with greater specific strength [1]. Belongs to the class of Advanced High-Strength Steels (AHSS), FeMnAlC steels have a high capacity to store dislocations which results in a high work-hardening rate and delay in the onset of plastic instability. Consequently, these steels exhibit an outstanding combination of strength and ductility [2]. Compared with the conventional steels, the lower density is mainly obtained by aluminum additions, which, together with the lower atomic mass, is responsible for the increase in the lattice parameter. However, aluminum also reduces the steel''s stiffness, limiting potential applications. In this context, the introduction of ceramic particles in the FeMnAlC matrix is an interesting strategy to minimize the negative effects associated with the Al, increasing the stiffness and further decreasing the density. The present work tailored FeMnAlC steels reinforced by TiB_2particles formed by in-situ reaction. The compositions were designed by thermodynamic calculations and the composites were processed by arc-melting and spray-forming routes. It was evaluated the microstructure, physics and mechanical properties.

Acknowledgments:
This study was financed by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001. São Paulo Research Foundation (FAPESP) for financial support grant 2020/05049-1 and 2021/11537-1.

References:
[1] S. Chen, R. Rana, A. Haldar, R.K. Ray, Current state of Fe-Mn-Al-C low density steels, Prog. Mater. Sci. 89 (2017) 345-391. https://doi.org/10.1016/j.pmatsci.2017.05.002. [2] A.L. Vidilli, L.B. Otani, W. Wolf, C.S. Kiminami, W.J. Botta, F.G. Coury, C. Bolfarini, Design of a FeMnAlC steel with TWIP effect and evaluation of its tensile and fatigue properties, J. Alloys Compd. 831 (2020) 154806. https://doi.org/10.1016/j.jallcom.2020.154806.

Design of Thermally Stable Microstructure After Equal Channel Angular Pressing at Cryogenic Temperatures

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Equal Channel Angular Pressing (ECAP) at cryogenic temperatures has been adopted to increase the strain severity by suppressing dynamic recovery and accumulating crystalline defects. However, severely deformed microstructures at cryogenic temperatures have limited use due to the microstructural instability presented, a consequence of the high internal energy stored as crystalline defects (vacancies and dislocations). In fact, some studies observed an evident grain growth after cryogenic processing when these materials were stored at room temperature. In this sense, this work aimed to design microstructures that are long-term stable after processing at cryogenic temperatures. The adopted strategy was to introduce a fine dispersion of precipitates in a severely deformed microstructure, to anchor the movement of dislocations and/or high and low angle contours. As a consequence, thermally activated phenomena, i.e., recovery and recrystallization would have their kinetics significantly reduced. Hence, the present work investigated the thermal and microstructural stability Cu-0.7Cr-0.07Zr alloy after processing by ECAP at cryogenic temperature, comparing it with electrolytic copper processed under the same conditions. After ECAP processing, the Cu-0.7Cr-0.07Zr alloy was subjected to aging treatment. The results showed that, after cryogenic processing, pure copper was microstructurally unstable, even when stored at room temperature, while the precipitates formed during aging were effective in stabilizing the microstructure of the Cu-0.7Cr-0.07Zr alloy.

The authors would like to express their gratitude to Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES - process nº 88882.332697/2010-01)

Development of a new reagent for the reveling of theprior austenite grain boundaries of AISI D2 steel.

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For the steels'' microstructure characterization, it is necessary to use a revealing solution. Previous research has shown that the reagents currently used to reveal the grain boundary also attack the interior of the grains, resulting in stains, and pitting corrosion, which makes it difficult to use an image analysis program to quantify them. The objective of the current work was to develop a reagent capable of revealing only the grain boundaries. Through the formulation of solutions based on Vilella''s reagent, it was possible to obtain a solution capable of exclusively revealing the primary austenitic grain contour without the use of a high-temperature chemical etching, thus allowing a better quality in the outline of the contours. The analyzed steel was AISI D2 steel, and from sanding, polishing, and subsequent chemical etching with the developed solution, it was possible to outline only the grain contour without generating pits of corrosion, or stains, with no need to use an image editing program to analyze the average diameter of primary austenitic grains.

Acknowledgment

The authors would like to thank the Department of Manufacturing Engineering and Materials at the Faculty of Mechanical Engineering at the State University of Campinas - DEMM/FEM/UNICAMP.

References

[1] **VANDER VOORT, G. F,** Revealing Prior-Austenite Grain Boundaries, Microscopy and Microanalysis 16(S2):774-775, 2010.

[2] **Brewer, A.W., Erven, K.A., Krauss, G.**, Etching and image analysis of prior austenite grain boundaries in hardened steels, Materials Characterization, Volume 27, Issue 1, 1991, Pages 53-56.

[3] **Faria, G. M., Pimenta, P. N.,** Avaliação da formulação de reativos à base de ácido pícrico visando a revelação do grão austenítico prévio em diferentes tipos de aços. Tecnologia em Metalurgia, Materiais e Mineração, 2021

DFT study and Hirshfeld surface analysis of a coordination complex of manganese(II) with 1,10-phenanthroline and maleate ligands

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The theoretical studies of coordination complexes were improved with the implementation of density functional theory (DFT) methods. Reliable DFT functionals to study coordination complexes have been developed [1]. The objective of this work was to investigate the geometrical, electronic, and energetic properties of a Mn(II) coordination complex with 1,10-phenanthroline and maleate ligands using the DFT and to study the main intermolecular interactions in the crystal lattice of the complex through Hirshfeld surfaces (HS) analyses. DFT calculations were performed with the Gaussian 16 software, using the PBE1PBE functional and the 6-311++G(d,p) basis set for H, O, C, and N atoms, employing the SDD pseudopotential and basis set for the Mn atom. HS analyses were performed using the CrystalExplorer software [2], starting from data available in a crystallographic database [3]. From the optimized geometry obtained from the DFT calculations, we observe that the complex has a distorted octahedral structure. The values of Gibbs free energy and enthalpy of the complexation are negative, indicating that the formation of the complex is spontaneous and exothermic. The HOMO and LUMO have been plotted and the energy gap between α-HOMO (the true HOMO) and β-LUMO is 4.19 eV, which was used for the calculation of reactivity indices. HS analyses indicated that H···H and H···O interactions arethe most significant, contributing with 36.5% and 35.3% of the intermolecular interactionsin the crystal, respectively. In this way, this study showed that the formation of the coordination complex is favorable and revealed the main interactions contributing to the stability of the crystal.

[1] G. R. Schleder, A. C. M. Padilha, C. M. Acosta, et. al., Jour. Phys. Mater., v. 2, p. 1, 2019.
[2] J. J. McKinnon, D. Jayatilaka, M. A. Spackman, Chem. Commun., v. 37, p. 3814, 2007.
[3] J. M. Yang, Z. H. Zhou, H. Zhang, H. et.al., Inorg. Chim. Act., v. 358, p. 1841, 2005.

DLC coatings applied in the wind turbines gearbox

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The demand for more sustainable alternative sources of energy production is growing all over the world. In this context, wind energy proves to be a valuable clean energy source to diversify the global energy matrix. Wind turbines are devices that convert the wind kinetic energy into electrical energy, with their main components being the blades and gearbox. The blades, fixed to the rotor, are responsible for capturing the wind and transmitting itspower to the rotor shaft, which then transfers the slow movement to the gearbox. Thegearbox transforms this rotation into a higher speed, which is responsible for generatingenergy in the stator. Soon, the gearbox is a critical part of this system and it is subject tocontinuous wear due to friction between metallic components. The wear of gears and bearings can lead to early equipment failure, increased maintenance costs, and productionbreakdown. In this context, the application of DLC coatings appears as a viable solution tomitigate it. In this study, DLC coatings are proposed for deposition on the steels surface toreduce the coefficient of friction and increase the resistance of the material. DLC will beevaluated in terms of their quality and stress state through Raman Spectroscopy. Additionally, the adhesion of the DLC to the steel substrate will be assessed usingscratching and Rockwell C tests. The morphology of the DLC coatings will be examinedusing a Scanning Electron Microscope. Moreover, the hardness of the DLC coatings will bedetermined by conducting nanohardness tests, while the wear resistance will be evaluatedthrough a tribometer test involving a spherical tip. DLC has high hardness, is chemicallyinert, resistant to wear, and reduces the surface roughness of steel. These DLC unique properties can provide the gearbox with less internal friction and greater durability contributing to increased wind turbine productivity.

EFFECT OF AGING AND COLD FORMING OF THE ALUMINUM ALLOY (AA6351)

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Aluminum alloys are versatile enough for its lightness, ductility and toughness when compared to steels. The handling of some alloying elements intentionally added in Al alloys gives great improvement in mechanical properties, ductility and toughness. Heat treatments when applied in optimum conditions make the alloys with even more efficient properties. With the solubilization followed by aging, we can achieve a considerable increase in resistance. The objective of this study was to determine the shortest time of aging with 100% cold forming and 0% cold forming in the AA6351 alloy. It was observed that no 0% cold forming samples showed an increase in microhardness when aged up to 150 min, while 100% the cold forming samples there were no significant differences up to 180 min. The microstructure of these conditions showed that the 0% cold forming samples did not present visible microstructural change, while in 100% cold forming samples there were the presence of deformation bands.

Effect of aluminum alloying on the structure and properties of TiNiCuNb shape memory alloy

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The microstructure, mechanical behavior and martensitic transformation (MT) were evaluated in regard to the effect of low Al (x=0, 0.5 and 1 at.%) content on the Ti48Ni38Cu(10-x)Nb4Alx (at.%) shape memory alloy (SMA). As cast alloys were subjectedto the X-ray diffraction (XRD) technique to analyze phase constituents and cristallinity; scanning electron microscope (SEM) to discover secondary phase particles sizes, morphology, homogeneity and distribution along the structure, and energy-dispersive X-ray spectroscopy (EDS) mapping to study the distribution of the chemical elements. The MT critical temperatures and enthalpy changes were determined by differencial scanning calorimeter (DSC). Furthermore, hardness measurements were used to evaluate the microstructure influence in the mechanical properties. Experimental results show that the increase of Al content is capable of successfully enhance microstructure refinement and homogeneity as observed by MEV and reduce MT temperatures. This newly developed alloy contributes to the development of SMAs with optimized microstructure for new applications, such as solid-state refrigeration systems.

Acknowledgements: This work was supported by Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq).

Effect of bismuth addition on the growth rates, cooling rates, Halo-like dendrite tertiary spacing and Rockwell hardness of the Al-12.6wt.%Si eutectic alloy

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Al-Si alloys are increasingly being employed in the aerospace industry, satellite bearings, and inertial navigation systems [1]. However, the inherent fragile nature of Si crystals represents a limitation to the development of new applications in automotive and aerospace industries, which requires good ductility [1]. It is well known that bismuth (Bi), as an alloying element, is widely used to refine the microstructure of aluminum alloys as the Al-Si alloy system [1,2]. In this sense, the main purpose of this work is to carry out an experimental study on the effect of bismuth addition on the growth rates (G_R), cooling rates (C_R), Halo-like dendrite tertiary spacing (λ_3) and Rockwell hardness F (HRF) of the Al-12.6wt.%Si eutectic alloy. To evaluate the effect of both G_R and C_R on dendritic microstructure, optical microscopy techniques and Image J software were used to measure the λ_3 values at positions from the heat transfer surface of a water-cooled directional solidification device. The mathematical expressions, given by the general equations $\lambda_3 = \text{Constant}(G_R)^{-1/4}$ and $\lambda_3 = \text{Constant}(C_R)^{-1/4}$ characterized the λ_3 variations as a function of the respective solidification thermal parameters. It was found that the addition of bismuth decreased the HRF and λ_3 values. It was also observed that the cooling system imposed a decreasing profile of λ3 values from the bottom to the top of the molten ingot as well as the HRF results decreased with increasing λ_3 for both alloys. A comparative analysis was also carried out with literature values.

Acknowledgements:

The authors are thankful for the structure and financial support provided by IFPA, UFPA, FAPESPA, CAPES, and CNPq for the development of this work.

References:
[1] T.A, Costa et al., The International Journal of Advanced Manufacturing Technology, vol. 105, 1391-1410 (2019)
[2] S. Farahany et al., Metals and Materials International, vol. 20, 929-938 (2014)

Effect of Cu additions on the wear behavior of AISI 316L single-walls manufactured by Plasma TransferredArc

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Modification of austenitic stainless steels with Cu induces a range of changes from thermal conductivity to biological resistance. This investigation further contributes to this discussion as it addresses the impact of Cu on the wear resistance of AISI 316L. Cu was added to the atomized stainless steel as microparticles (MPs) and as nanoparticles (NPs). Cu nanoparticles (NPs) have been used to enhance the properties of bulk materials due to their high surface-to-volume ratio [1, 2]. The impact of the NPs depends on the processing technique that will determine their distribution on the microstructure and hence on the properties. In this study, plasma transferred arc (PTA) was used to fabricate AISI 316L single-walls with and without Cu. 1 and 5 wt% Cu microparticles (MPs), 1 wt% Cu NPs and1 wt% CuO NPs were individually added to the gas-atomized stainless steel. Deposited single walls were subjected to solubilization at 1100 ºC for 0.5 h followed by air quenching. Aging treatment was carried out at 700 ºC for 0.33, 1.0 and 3 h. As built and heat-treated microstructures were assessed by optical microscopy and hardness measurements. 100Cr6 ball-on-disk wear tests were performed at room temperature under an applied force of 5.0 N, rotation of 125 rpm and 2500 laps. Wear tracks were evaluated by confocal microscopy and scanning electron microscopy (SEM). The hardening effect due to Cu precipitation was observed in all samples containing Cu, whereby longer aging times led to higher hardness. Samples containing CuO NPs showed the highest hardness in all heat-treated conditions. Regardless of the heat treatment applied, a higher Cu content led to a slightly lower coefficient of friction (COF) which resulted in a significant reduction in sample wear rate (SWR). The presence of NPs had no significant impact on COF or on SWR.

References:

[1] G.S. Prass, A.S.C.M. d' Oliveira. Surf. Coat. Technol., vol. 461. 2023
[2] Y.N. Slavin et al. J. Nanobiotechnol., vol. 15, p. 65. 2017

Effect of current density on obtaining the Fe–Mo–Palloy by the electrodeposition process

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The study of the addition of molybdenium by electrodeposition in alloys containing less noble metals such as iron can improve their mechanical properties and corrosion resistance [1]. In addition to the addition of iron, other elements such as phosphorus can improve the mechanical properties and corrosion resistance of iron and molybdenum-based alloys [2]. Thus, the objective of this work was obtain the Fe-Mo-P alloy from the electrodeposition process and characterize the properties of the coatings. An electrolytic bath composed of sodium citrate (0.50 M), iron sulfate (0.03 M), sodium molybdate (0.09 M) and sodium hypophosphite (0.09 M) was developed. The parameter evaluated in the deposition process was the current density (30 mA/cm2 and 60 mA/cm2), at pH 6. The chemical composition was evaluated by EDX, the microstructure by XRD and the surface morphology by SEM. The corrosion resistance of the coatings was evaluated using the Potentiodynamic Polarization (PP) technique, in a saline medium (NaCl, 3.5%). The EDX results confirmed the formation of the Fe-Mo-P alloy following the induced co-deposition mechanism [3]. SEM results showed the presence of microcracks and the formation of superficial nodules. The XRDresults confirmed the amorphous character of the coatings. The corrosion results showedthat the coating obtained at 60 mA/cm2 and pH 6 obtained the noblest corrosion potential(-0.5 V) and the highest polarization resistance value (20.00 Ω.cm2), presenting, therefore the highest resistance to corrosion.

Effect of EBW process welding parameters on AISI304L stainless steel bead geometry

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One important field in the welding researches is the study of the relationship between the welding bead geometry and welding machine parameters. In electron beam welding (EBW) process, the fine controlled parameters enable a detail understand of the weld profile. EBW provides a concentrated beam of energy which can result in a high ratio of weld depth to bead width, which can be called a keyhole. EBW is widely study because of its importance in nuclear and aerospace application, mainly as a result of keyhole bead geometry. The relationship between the welding bead geometry, width root and depth with welding machine parameters is presented in this work by setting beam current and beam deflection frequency. All experiments were carried out on samples of AISI 304L alloy with a thickness (t) = 10 mm, accelerating voltage U = 60 KV, beam current range I = 34 - 43 mA, welding speed v = 480 mm/min, vacuum p < 10-4 mbar, beam deflection width d = 1.2 mm and beam deflection frequency f = 400 / 600 Hz. The weld joint geometry has changed by varying of beam current and beam deflection frequency. The weld penetration was increased by increment of beam current and deflection frequency. Set the frequency at f = 600 Hz and changing the beam current from 34 mA to 39 mA, it was observed that penetration rise from 7.38 mm to 8.46 mm, respectively. On the other hand, when was modified deflection frequency from 400 Hz to 600 Hz, the average welding penetration surged 6.3%. Moreover, with deflection frequency kept in f = 400 Hz, a beam current increment from I = 37 mA to I = 43 mA led to grow the welding penetration from 7.56 to 8.63, respectively. The results of weld width root showed no variation due to an increase of beam current, however applying a change of electron beam deflection frequency from 600 Hz to 400 Hz was verified a slight increase in it. In conclusion, the welding bead geometry is directly affected by varying of current beam and deflection frequency parameters.

EFFECT OF HEATING RATE ON THE KINETICS OF MARTENSITIC TRANSFORMATION OF NI-TI-HF ALLOYS: A STUDY USING DIFFERENTIAL SCANNING CALORIMETRY (DSC)

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There are some alloys with shape memory of the Ni-Ti system considered as HTSMA (High Temperature Shape Memory Alloys). This occurs because a third element (Ni-Ti-X) is added to them, which, depending on the processing used, can obtain martensitic transformation temperatures (MS) above 100°C. Examples of these alloys include: Ti-Ni-Pd, Ti-Ni-Ta, Ti-Ni-Au, Ti-Ni-Hf. Hf is a more economically viable chemical element among those cited as thethird component of these ternary alloys. Thus, it was chosen for this work because of the possibility of these alloys being applied as a sensor/actuator in systems where martensitic transformation temperatures above 100°C are required. The $Ni_{50}Ti_{36}Hf_{14}$ (%at) alloy was prepared in an arc melting furnace. The transformation kinetics analyzes were carried out in a Differential Scanning Calorimeter (DSC) with heating rates of 2.5 °C/min, 5°C/min, 10°C/min and 20°C/min in the range of temperature between -30°C to 400°C and the startand end temperatures of the transformation varied according to the composition of thealloy. The results obtained for martensitic transformation (MS) temperatures during heatingconfirmed the Kolmogorov-Johnson-Mehl-Avrami Model (KJMA) pattern for a process mediated by nucleation and growth. Transformation enthalpies increased as heating ratesincreased.

Effect of Mg addition on the primary and secondary dendritic spacing of Al-4.5wt.%Cu alloy

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Understanding the solidification process in manufacturing metal parts and components seeks to optimize structural characteristics and, consequently, the performance and quality of the product obtained. The dendritic microstructure found in solidified alloys is a consequence of the instability of the solid-liquid interface during phase change and directly affects the thermal and mechanical properties of materials [1,2]. Associated with this, chemical elements added to an alloy also influence the thermophysical characteristics of the material obtained. The Al-Cu system, especially with the copper content ranging from 2% to 5%, offers high mechanical strength and hardness when subjected to heat treatments. The low specific weight, allied to such properties, has made this torque a very interesting alternative in the manufacture of products for the automotive and aeronautic industries, such as cylinder heads and engine blocks. Adding Mg to Al strengthens the alloy not only through a solid solution strengthening, but also through a substantial dispersion strengthening mechanism through the formation of Al_2Cu and Al_2CuMg phases by heat treatment [3]. The main purpose of this work is to investigate the effect of Mg on primary and secondary dendritic arm spacings, respectively λ_1, λ_2, of an Al-4.5wt%Cu alloy horizontally solidified under transient conditions. The obtained results show significant changes between the observed microstructures of studied alloys, i.e., the addition of 1% Mg increased the $\lambda_1$values in Al-4.5wt%Cu-1wt%Mg as well as second phase particles are formed according to MEV.

[1] M. Yang, S.-M., Xiong, Z., Guo. Acta Materialia 112, 261-272 (2016)
[2] T. Isensee, D. Tourret. Acta Materialia 234, 118035 (2022)
[3] M. Zamani, S. Toschi, A. Morri, L. Ceschini, S. Seifeddine. Journal of Thermal Analysis and Calorimetry 139, 3427-3440 (2020)

Effect of Nb and Ti additions on phases, microstructure and hardness of equimolar AlCoCrFeNi high-entropy alloy

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The AlCoCrFeNi is one of the most investigated and promising high-entropy alloy (HEA) systems, due to its excelent synergy between phases control composition and properties [1]. The properties of this alloy can be improved by the addition of a sixth element in this system, among which the transition metals such as Cu, Mo, V, Nb and Ti are the most remarkable [2]. The aim of this work was to study the effect of Nb and Ti additions on the phases constitution, microstructure and hardness of equimolar AlCoCrFeNi HEA. The CALPHAD method using Thermo-Calc® software was employed for to compare and investigate the phases predicted by thermodynamic calculations with those obtained experimentally in ingots produced by arc melting. The phases and microstructure of as- casted samples were characterized by X-ray Diffraction (XRD) and by Scanning ElectronMicroscopy with Energy Dispersive Spectroscopy (SEM-EDS). The Vickers Hardness (HV) map and nanoindentation were performed and the results were related with the microstructure. The results of XRD and SEM-EDS analyzes agreed with the Thermo-Calc calculations, showing that the alloys are composed by solid solutions formed at high temperatures, presenting the ordered BCC-B2 as dominant phase. Nb additions leads to the formation of the Laves phase, while increasing the amount of Ti leads to the formation additional complex phases resulting from the BCC-A2 decomposition. The lattice parameter of the alloys increased with de Nb and Ti additions, due to the large atomic radius of these elements, which consequently result in a increase in hardness by the solid solution streghthening mechanism. The hardness of the AlCoCrFeNi alloy increased from 461 HV to 591 HV and 790 HV with the addition of Ti and Nb, respectively.

References:

[1] A.M. Zemanate, A.M.J. Júnior, G.F. de Lima Andreani, V. Roche and K.R. Cardoso. Electrochimica Acta, 141844, (2023).

[2] M. Löbel, T. Lindner, T. Mehner and T. Lampke. Entropy, 20 (7), 505, (2018).

Effect of solution and aging processes on the microstructure and wear resistance of a horizontally solidified AlCuNb alloy

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Niobium plays an important role in the production of metallic alloys due to its high melting point, resistance to acid attacks and superconductivity at elevated temperatures [1]. This study analyzed the effects of the solidification and heat-treating parameters on the microstructure and the wear resistance of the Al-3wt.%Cu-5wt.%Nb. To achieve this, the alloy was obtained via unsteady-state solidification using a water-cooled horizontal solidification device. The investigated solidification variables were the growth and cooling rates ($V_L$and T_R, respectively). Samples obtained from the resulting ingot were analyzed both in the as-cast state as well as after being submitted to a T6 heat-treatment (T6-HT) which consisted of 3 hours of solution treatment at 490°C and 3 hours of artificial aging at 155°C. It was found that there were no significant changes in morphology of both the niobium andAl_3Nb before and after the T6-HT, however, it was able to effectively remove the copperfrom the interdendritic and intergranular regions and precipitate it inside the aluminum matrix, which grants the alloy greater resistance. The only way through which copper remained in such regions was when it combined with trace amounts of iron left over fromthe aluminum production process, as well as with niobium, forming intermetallic phases insusceptible to the heat-treatment. Such greater resistance was evidenced by the wear tests results, which showed that the T6-HT was ultimately able to decrease the wear rate ofthe investigated alloy by around 30% when compared to its as-cast state.

Acknowledgements:

The authors acknowledge the financial support provided by IFPA, UFPA, and CNPq - National Council for Scientific and Technological Development (Grant 304924/2020-2).

References:

[1] MENDES, Marcio Willians Duarte. Obtenção de pós de Nb a partir da redução aluminotérmica com ignição por plasma. Dissertação - Universidade Federal do Rio Grandedo Norte, Natal, 2006.

Effect of sputtering pressure on the physical propertiesof Zr-based metallic glass thin films

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Amorphous metals, also known as metallic glasses, are materials that exhibit good thermal and electrical conductivity, corrosion and wear resistance [1], and a high Young's modulusdue to their lack of lattice structure. As thin films, these materials can be very interestingfor coatings [2], micro-electromechanical systems [3], and flexible strain sensors [4], amongother potential applications. We are investigating the preparation and properties of Zr- based metallic glass thin films grown by sputtering, for coatings and wearable sensors. Wewill discuss the effect of the Ar pressure during deposition - the working pressure - on thephysical properties of thin films from targets of Zr67Fe33 and Zr55Cu30Ni5Al10 alloys. Thethin films, 200 nm thick, were prepared using DC magnetron sputtering and at chamberworking pressures of 0.4, 0.63, and 1.0 Pa. It is expected that these conditions induce structural and morphological changes in the samples that result in modifications in theirproperties. The obtained films were characterized by X-ray diffraction, X-ray fluorescence, X-ray photoelectron spectroscopy, scanning electron microscopy, 57Fe Conversion electron Mössbauer spectroscopy (Zr67Fe33), and electrical measurements. The corrosion resistance was assessed through potentiodynamic polarization curves. The influence of the working pressure on the microstructure, corrosion behavior, and electrical properties of the films will be presented and discussed.

Acknowledgments:Financial support from CNEN, CNPq, CAPES, and FAPEMIG is gratefully acknowledged.

[1] W. Wang, H. Mraied, W. Diyatmika, J. P. Chu, L. Li, W. Cai. Surf. and Coat. Technology 402, 126324 (2020).
[2] S. T. Rajan, A. Arockiarajan. Jour. of Alloys and Compounds 876 (2021).
[3] P. Yiu, W. Diyatmika, N. Bönninghoff, Y.-C. Lu, B.-Z. Lai, J. P. Chu, Jour. of Applied Physics 127 (3), 1 (2020).
[4] Y.-C. Lu, C.-Y. Chiang, Y.-C. Chen, Y.-C. Lin, T. Ono, Y.-C. Tsai, Jap. Jour. of Applied Physics (2020), Vol. 59, pp. 1.

Effect of the hydrolysis time on the corrosion resistance of AA2024-T3 anodized in TSA and sealedwith hybrid coatings

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Chromium-based anodizing and posttreatment steps are usually employed in the aircraft industry. However, despite offering good corrosion resistance and self-healing properties, as chromate compounds are toxic to health and the environment, chromium-based surface treatments will be prohibited in the aerospace industry soon. Tartaric-sulfuric acid (TSA) anodizing is a promising environment-compliant alternative, which is already being used at the industrial level. However, the corrosion resistance of the anodized layer alone is not sufficient to guarantee the required safety levels for an aircraft and more complex systems should be employed. In this investigation, AA2024 T3 specimens were anodized in TSA and subsequently sealed by a hybrid sol-gel coating (tetraethoxysilane (TEOS) and 3-glycidoxypropyltrimethoxysilane (GPTMS)), which hydrolysis solution was aged for different times. The morphology of the sealed anodic films and their thicknesses were evaluated by field emission scanning electron microscopy (FE-SEM) and Rutherford backscattering spectroscopy (RBS). The aging of the hydrolysis solution was followed by FTIR spectroscopy. The corrosion resistance of the samples was evaluated by electrochemical impedance spectroscopy (EIS) and salt-spray tests. FE-SEM characterization showed good coverage of the anodized surface by the hybrid layer. EIS experiments demonstrated improved corrosion resistance for increased hydrolysis time, and good responses were obtained for up to six months of aging. The relatively long shelf life of the hydrolysis solution is a good prospect for future industrial use of the hybrid coating.

Effects of growth and cooling rates, and secondary dendritic spacing on the electrochemical corrosion resistance of an AlCuNb alloy

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Al-Cu alloys are generally less corrosion resistant than other alloys. In these alloys, intermetallics of Al_2Cu, Al2CuMg, and Al_7Cu_2Fe are some of those responsible for initiating galvanic corrosion with the matrix [1]. Niobium at room temperature resists well to the action of hydrochloric (up to 35%), sulfuric (up to 95%), concentrated nitric, phosphoric, chromic, acetic, formic and citric acids. It has resistance to corrosion due to the formation of a superficial oxide film responsible for its passivation [2]. In this sense, the main objective of this work is to evaluate the effects of solidification thermal parameters (STP) and the length of the microstructural scale, such as growth and cooling rates (VL and TR), and secondary dendritic spacing (λ_2) on the corrosion resistance (CR) of horizontally solidified Al-3Cu-0.5Nb alloy (wt.%) using a water-cooled device. Electrochemical measurements of corrosion potential (Ecorr) variations with time and potentiodynamic polarization curves were obtained in a 0.2 M HCl electrolyte (25 ºC). The corrosion tests were carried out attwo positions in relation to cooled interface of the studied alloy (10 and 70mm). The results indicated that in the position 70 mm of the cooled wall the corrosion density (Icorr) increase and shows minimum shift of the Ecorr in the negative potential direction. This work demonstrates that the $\lambda_2$is a very important factor to influence CR.

Acknowledgements:
The authors acknowledge the financial support provided by Federal Institute of Education Science and Technology of Pará (IFPA/PPGEMat), Federal University of Pará (UFPA/ITEC/PPGEM), and CNPq - National Council Scientific and Technological Development (Grant 304924/2020-2).

References:
[1] BARROS, A., CRUZ, C., SILVA, A. P., CHEUNG, N., GARCIA, A., ROCHA, O., MOREIRA, A. Acta Metall. Sin., vol. 32, pp. 695-709, 2019.
[2] SOUSA, R., FERNANDES, L. E., GUERRA, W. Nióbio, Revista Química Nova na Escola. Vol. 35, n1. pp. 68-69, 2013.

Electrochemical behavior of Cu-9Al-2Mn-xSn alloys

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Copper-rich Cu-Al-Mn alloys are widely studied due to their mechanical and thermomechanical properties. The presence of Mn in these alloys, combined with low content of aluminum, improves the ductility and increases the temperature at which martensitic transformation occurs. Moreover, Cu-based alloys show corrosion resistance that allows the application in industrial devices such as suction pumps and in naval industry[1-2]. In this study, different Sn contents were evaluated in the Cu-9%Al-2%MnX%Sn alloys(X = 0.5, 1, 2, 4, and 6). The corrosion resistance in 0.5 mol L^{-1}NaCl of the aforementionedalloys, as well as the relationship between this property and the phases composition, werestudied using X-ray diffraction, optical microscopy, cyclic voltammetry, open circuit potential, and potentiodynamic polarization. The results showed that there is a grain refinement when the Sn content increases. In addition, at higher concentrations than 2%Sn, a new phase is formed in the alloys. Corrosion potentials became more positive with theSn addition, but the corrosion current densities increased up to 1.0% Sn and then decreasedat higher concentrations. This suggests that the initial increase in tin content impairs the corrosion resistance of the materials, which may be related to the formation of new phasescontaining this element. Further increase in tin content reduced the quantity of phases inthe alloy. There is evidence that the presence of tin modifies the well-known copper oxidation mechanism in a 3.5% NaCl solution.

Acknowledgments:The authors thank FAPESP, CNPq, and CAPES for the financial support.

References:

[1] M. A. Shaik, K. H. Syed, and B. R. Golla, “Electrochemical behavior of mechanically alloyed hard Cu-Al alloys in marine environment,” Corros Sci, vol. 153, pp. 249-257, Jun. 2019.

[2] Serafim, Lucas Soares et al. “Microstructural characterization and mechanical properties of Cu-Al-Mn-Ti-B alloy susceptible to shape memory effect”. 2019.

Electrochemical corrosion behavior of SAE 300M and dual-phase DP600 steels laser-welded after heat treatments

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In the present study, the electrochemical corrosion behavior of laser-welded dissimilar steel joints was evaluated. SAE 300M is an ultra high strength steel and used in the aerospace industry due to its high resistance [1]. Dual-phase DP600 belongs to the grade of advanced high-strength steels whose development resulted from the automotive industry's need for low-cost sheet material [2]. The fiber laser welding process was employed as this type of welding is faster and cleaner than other processes, with minimal change to the steel's microstructure. The welding parameters applied were 1800 Watts of power and 50 mm/s of speed to join the SAE 300M and DP600, with a sheet thickness of 2.7 and 2.2 mm, respectively. After welding, the welded joints underwent three types of heat treatments: tempering, quenching and tempering, and intercritical quenching. Potentiodynamic polarization measurements were performed in a 3.5 wt.% NaCl solution at 25 °C to evaluate corrosion behavior of the welded joints. The findings about corrosion behavior revealed that the Fusion Zone (ZF) is more prone to suffer the effects of corrosion than steels surrounding this region, either in 300M or DP600 steel. For untreated welded joints and tempering treatments, the rate registered for annual corrosion is low with little change in mass loss and welded joint's lifetime, being 0.44 mm per year for untreated condition and 0.60 mm per year for tempering condition. For the intercritical tempering, tempering and tempering treatments, the value was more relevant, the corrosion rate was higher, the mass loss was around 10 fold higher than the other conditions, reducing the useful joint’s lifetime.

Acknowledgments: This research was carried out with the support of CAPES and members of the Mechanics group at ITA.

References:

[1]A. Bag; D. Delbergue; P. Bocher; M. Lévesque; M. Brochu. Int. J. Fatigue, vol.118, 2019
[2]G.A.W. Sweet; I.W. Donaldso C.T. Schade; M.Y. Amegadzie; D.P. Bishop. Add. Manuf., vol.47, 2021

Empowering Professionals through Microscopy Training: Outcomes and Impacts of the LCE/UFSCarMicroscopy School

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The Microscopy School is a specialization course offered by the Structural Characterization Laboratory (LCE), of the Materials Engineering Department at UFSCar. This course is annually offered, and its main results, impacts and outcomes will be showcased. The course is being offered for more than 20 years and is attended by professionals from engineering, physics, chemistry, and biological sciences fields. The Microscopy School provides training on several microscopy techniques, including transmission and scanning electron microscopy as well as other characterization techniques such as x-ray diffraction. Participants receive hands-on training on sample preparation, instrument operation, and data analysis, which enhance their understanding of material structure and properties. The course was reported to be highly beneficial to undergraduate and graduate students, as well as researchers from industries and research centers in Brazil. The Microscopy School offered participants the opportunity to acquire state-of-the-art knowledge and skills, which they could apply in their research projects, contributing to the advancement of the scientific and technological development of the country. The training also promoted the transfer of knowledge and technology between academia and industry, which is essential for solving real-world problems. A summary of the main activities performed during the course as well as summaries of reviews of the course will be presented. The structure of the course will be showcased and the strategy used to be a useful course for both academia and industry. In conclusion, the Microscopy School offered by the LCE of UFSCar is being successful in providing quality training to professionals from different fields, promoting knowledge exchange and enhancing the scientific and technological development of the country.

Enhanced electronic transport properties of Te roll-like nanostructures.

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In this work, the electronic transport properties of Te roll-like nanostructures were investigated in a broad temperature range by fabricating single-nanostructure back-gated field-effect-transistors via photolithography. These one-dimensional nanostructures, with a unique roll-like morphology, were produced by a facile synthesis and extensively studied by scanning and transmission electron microscopy. The nanostructures are made of pure and crystalline Tellurium with trigonal structure (t-Te), and exhibit p-type conductivity with enhanced field-effect hole mobility between 273 cm^2/Vs at 320 K and 881 cm^2/Vs at 5 K. The thermal ionization of shallow acceptors, with small ionization energy between 2 and 4 meV, leads to free-hole conduction at high temperatures. The free-hole mobility follows a negative power-law temperature behavior, with an exponent between −1.28 and −1.42, indicating strong phonon scattering in this temperature range. At lower temperatures, the electronic conduction is dominated by nearest-neighbor hopping (NNH) conduction in the acceptor band, with a small activation energy $E_{NNH} \approx 0.6$ meV and an acceptor concentration of $N_A \approx 1 \times 10^{16} cm^{-3}$. These results demonstrate the enhanced electrical properties of these nanostructures, with a small disorder, and superior quality for nanodevice applications.

Published: Beilstein J. Nanotechnol. 2022, 13, 1284-1291. https://doi.org/10.3762/bjnano.13.106

Evaluation of citronella essential oil as a corrosion inhibitor for carbon steel and a copper alloy exposed to soybean biodiesel

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In recent years, world population growth has generated a great demand for new technologies that can serve strategic areas such as energy, healty, food, transport. Regarding the energy matrix, there is a movement that looks for alternatives to replace the energy matrix based on fossil fuels. In this sense, biofuels are considered a good alternative, mainly because they present an environmentally friendly behavior [1-2]. Among the different types of biofuels, biodiesel is the main option to replace fossil diesel, however, biodiesel has disadvantages such as being susceptible to oxidative degradation, more hygroscopic and generally more corrosive than diesel [1-4]. Considering the high costs of corrosion and the greater corrosivity of biodiesel, research has been carried out to understand the behavior of different metallic alloys exposed to biodiesel and to evaluate compounds that act as antioxidants, maintaining the stability of biodiesel and reducing corrosion [1-4]. This work evaluated the effect of adding citronella essential oil on the corrosion inhibition of two metal alloys: carbon steel and copper alloy in contact with soybean biodiesel. Immersion tests at temperature of 25°C were carried out and made it possible to calculate the corrosion rate of the metallic alloys and the efficiency of inhibition of the essential oil. It was observed that the essential oil acts as a corrosion inhibitor for both carbon steel and copper alloy, however the effect was more significant for steel.

Acknowledgements:
FAPERJ, CNPq and UERJ for financial support.

References:

[1] DEYAB, M. A., et al. Industrial Crops Products, 130, 146 - 150, 2019.
[2] FERNANDES, D.M., et al. Renewable Energy 139, 1263 - 1271, 2019
[3] JAIN, S., et al. Fuel 289, 119906, 2021.
[4] BAENA, L.M. and CALDERON, J.A., Heliyon, 6, e03735, 2020.

EVALUATION OF DEFORMATIONS OBTAINED IN THE TENSILE TESTING USING CORRELATE GOM SOFTWARE

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This study consisted of the implementation of the Digital Image Correlation (DIC) method, using the GOM CORRELATE 2D software provided by the German company GOM [1]. This application allowed the realization of more simplified, accurate, true, low-cost and easy-to-implement analyses, consistent with the state of the art for these applications in companies that work with material stamping. The ease of the technique allowed the use of cameras found on smartphones, to survey the deformations that occurred during the tensile test, in a controlled manner on the specimens. To carry out the tests and validate the technique, a high-strength cold-rolled steel was used, applied in the segment of auto parts, where samples were taken in the transverse direction of the lamination, and they were made in accordance with the ASTM A 370 standard [2]. Afterwards, layers of spray paint were applied, the first being white and then drying, a black layer with the intention of creating a random pattern, but uniformly on the surface of the specimen for the uniaxial tensile test. The tests were carried out in the EMIC equipment model DL 10000 of 100kN. The images obtained sequentially during the traction test of the specimen, previously painted, were loaded into the software and, after configurations and parameter adjustments, generated graphic images with value scales. The regions where the greatest deformations occurred in the tensile test were shown through the algorithms of the Digital Image Correlation technique to describe the behavior of the material. The product of this work was the elaboration of a procedure for using the GOM CORRELATE 2D software in the UniFOA laboratory.

Acknowledgements:

The authors are grateful to the UniFOA.

References:

[1] GOM Correlate. https://www.gom.com/en/products/zeiss-quality-suite/gom-correlate-pro.

[2] ASTM A370, Standart Test Methods and Definitions for Mechanical Testinhg of Steel Products, ASTM International, 2007.

Evaluation of electrical pulses technique as a self-healing mechanism in the AA5083 alloy applied in structural elements of the oil industry.

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The self-healing mechanism through electrical pulses is an effective alternative to promote the regeneration of microstructural defects in offshore oil industry metallic structural materials. It assists in atomic diffusion, stress relief, dislocation movement, microcrack closure and even recrystallization in some cases [1-2]. The effect of electrical pulses was analyzed on specimens of the AA5083-H112 alloy with a cross section of 15 mm^2, which were submitted to tensile tests to promote microstructural defects and then submitted to self-healing. Defects were generated with a pre-strain of 80% of the rupture strain. The pulses were emitted in closed circuit with an adapted welding source, model DigiPlus A7 600 in pulsed TIG mode. The peak current was 400A and base current 0A, with periods of 0.1s each. The complete pulse emission time ranged from 0s, 7s and 15s. The samples were resubmitted to tensile tests and evaluated. There was a small thermal effect reaching up to 142.50 ºC. The application of a current density of 26.67 A/mm^2proved to be effective in promoting an increase in ductility. The resulting deformations in the pre-deformed samples, after 0s, 7s and 15s of electrical pulses was 7.60%, 9.02% and 11.02%, respectively, indicating a significant gain in ductility. The microstructural characterization via SEM corroborated the evidence of the effect that occurred on the observed microstructural defects.

Acknowledgements:
To UFRN and to the Human Resources Program in Nanotechnology and New Materials Applied to the Petroleum, Natural Gas and Biofuels (PRH 32.1) and the NATIONAL PETROLEUM AGENCY, NATURAL GAS AND BIOFUELS (ANP).

References:
[1] CONRAD, Hans. Materials Science and Engineering: A, [S.L.], v. 287, n. 2, p. 227-237, Aug. 2000. Elsevier BV. http://dx.doi.org/10.1016/s0921-5093(00)00780-2.
[2] GRABOWSKI, Blazej; TASAN, C. Cem. Self-Healing Materials, [S.L.], p. 387-407, 2016. Springer International Publishing. http://dx.doi.org/10.1007/12_2015_337.

Evaluation of influence of Phosphatization on Electrodeposition of Nickel Coatings on CopperSubstrate

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Copper is widely used in the electricity and civil construction sectors, however it is susceptible to corrosion when exposed to aggressive environments [1]. Phosphatization pretreatment is a method that has been employed on various metal substrates to delay the corrosion process and improve the adhesion of coatings to the substrate through the formation of a porous layer of insoluble precipitates [2]. Phosphated and coated steel surfaces has been addressed in several studies, but investigation of protective methods for copper substrates is not widespread. Electrodeposited nickel coatings have been widely used due to their excellent properties to increase corrosion resistance, low-cost, high-efficiency and easy-to-perform process [3]. The aim of this study was to evaluate the influence of phosphatization on electrodeposited nickel coatings on copper substrates. For this, surface characterizations were performed under four conditions: uncoated substrate (C00), phosphatized substrate (C00F), substrate coated with electroplated nickel (C10), and phosphatized substrate subsequently coated with electroplated nickel (C10F). Morphological analysis of the obtained surfaces was performed through SEM and qualitative elemental composition was determined through EDS. Wear resistance was determined using a linear module tribometer with a 1 N load. Corrosion resistance properties were analyzed by potentiodynamic polarization test in 3.5% NaCl solution. The prior phosphatization of the surface resulted in a nickel coating with more lamellar structures on a smaller scale, showing improved corrosion and wear resistances.

Acknowledgments:
FINEP, CAPES, CNPq, FACEPE PROPESQI-UFPE, LBC-COMPOLAB/UFPE, LITPEG/UFPE and INTM/UFPE.

References:

[1] Panagopoulos, C.N. and Georgiou, E.P., Tecnol. Res. 8-1,125 (2011).
[2] Rubi, G. and Modi, O.P., Surface and coatings. Tecnol. 201-1866 (2006).
[3] Yu, B. and Zhu, G., Journal of Electroanalytical Chemistry 910, 116146 (2022).

Evaluation of metal oxide nanoparticle films on steel corrosion: selection of important variables

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The use of nanoparticles has been contributing to a technological advance in the industrial processes, as in the case of corrosion protection. In this sense, the objective of this research is to investigate the effect of nanoparticles of SiO2, TiO2, ZrO2, and their mixtures as inhibitors of the corrosion process of 1020 steel, using thin films obtained by dip coating. Dip-coating technique has been widely used in metallic surface coating processes with film formation that lead to corrosion protection [1]. In general, the films are obtained with: a solution withdrawal speed of 10cm/mi three immersio film drying temperature of 60°C for one hour, between each immersions and using distilled water as solvent in the dispersion of nanoparticles. The time of immersion has not being shown as an important variable. However, the temperature for the thermal treatment of the films at various times affects the corrosion inhibition, normally being tested at 500°C and 800°C with 1h and 2h [2-3]. Another relevant aspect is that each solution composition (concentrations and components) leads to different inhibition effects, also verifying that the mixture and proportion between components are also of extreme importance. In general, can be checked mixtures with 70%SiO2 and 30% TiO2 or ZrO2. In order to verification the corrosion rate are employees open-circuit potential tests and polarization curves. Considering these aspects of the literature, this work seeks to apply factorial planning as a tool to evaluate the best experimental condition to corrosion inhibition using SiO2, TiO2, ZrO2 components, of the fims obtaindeby dip coating.Acknowledgements: CAPES, PPGQ/UFJReference: [1] S. KIRTAY, J. Mat. Engineering and Performance, 23, 4309-4315, 2014. [2] M. Atik, P. De Lima Neto, M. A. Aegerter and L. A. Avaca, J. Appl. Electrochem., 25, 142-148, 1995. [3] P. de Lima Neto, M. Atik, L. A. Avaca and Michel A. Aegerter, J. Sol-Gel Scie. and Techn., 2, 529-534, 1994.

EVALUATION OF MURUMURU BARK POWDER AS CORROSION INHIBITORS FOR CARBON STEEL INACID MEDIUM

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Corrosion is a spontaneous process of deterioration of metals that is very common on a daily basis, especially in coastal regions. This fact causes great economic and environmental damage to society, then there is a need to use corrosion inhibitors. As a sustainable and economically viable alternative, natural corrosion inhibitors extracted from plants appear. This work aims to study the efficiency of murumuru bark powder as a corrosion inhibitor on SAE 1008 carbon steel in 1M HCl acid medium. To evaluate the inhibition efficiency, electrochemical impedance spectroscopy (EIS), gravimetric techniques and polarization curves were performed. Fourier Transform Infrared Spectroscopy (FTIR) was performed for chemical characterization. Through the results of the EIS it was observed that the presence of the inhibitor confers protection against corrosion and an efficiency of 78.2% for the concentration of 1.77 g.L-1. Analyzing the polarization curves, murumuru bark powder is an inhibitor that presents a mixed behavior [1]. The results of the gravimetric tests corroborate the electrochemical tests when they present an efficiency of 66.2% at the concentration 1.77 g.L-1. The FTIR assay showed the presence of hydroxyls and other functional groups in the inhibitor structure that may be responsible for inhibiting this corrosive process. In this way, it can be inferred that the Murumuru bark presents an inhibitory characteristic of corrosion in acidic medium in carbon steel SAE 1008.

Acknowledgements: FACEPE, FAPESB.

References: [1] A. M. Santos, I. P. Aquino, F. Cotting, I. V. Aoki, H. G. de Melo, V. R. Capelossi, Evaluation of Palm Kernel Cake Powder as Corrosion Inhibitor for Carbon Steel in Acidic Media, The Korean Institute of Metals and Materials, 2020.

Evaluation of steel alloys applied in roller presses for comminuting ores

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Reducing manufacturing costs is a crucial goal for the metallurgical sector, and one approach is to replace a material with a more affordable alternative that is equally or more effective. This study aims to investigate the feasibility of substituting a specific material, used in comminution rollers, with a large-scale produced tool steel. The materials studied include the current roller mill steel, 18NiCrMo14-6, and the modified H11 tool steel, both of which underwent equal heat treatment procedures. Normalized at 920°C, followed by a temper at 600°C, quenched in water and again tempered at 540°C. The primary distinction between the alloys is the reduction in carbon and nickel content, which is prevalent in the current steel, compensated by an increase in chromium, vanadium, and molybdenum content in the tool steel [1]. The microstructural analysis, performed using SEM and optical microscopy, revealed that both materials possess bainitic and martensitic structures despite having different alloying elements [2] [3]. The hardness values of the two materials were similar, with around 40 HRC for each. Finally, the tensile and impact tests demonstrated that the tool steel is a feasible substitute.

Acknowledgements: I express my gratitude to the Casemiro Monte Negro Filho Foundation for their valuable support towards the research conducted

References:

[1] MESQUITA, R. A.; BARBOSA, C. A. NOVO AÇO FERRAMENTA DE ALTA RESISTÊNCIAA QUENTE Tecnologia em Metalurgia e Materiais, São Paulo, v.3, n.3, p. 63-68, jan.-mar. 2007

[2] SHACKELFORD, J. F. MATERIAIS PARA ENGENHARIA. Ciência dos Materiais. São Paulo: Pearson Prentice Hall, 2008. p. 9. 3.

[3] BHADESHIA, H. K. D. H.; THE BAINITE TRANSFORMATION: UNRESOLVED ISSUES. Materials Science and Engineering: A, v. 273-275, p.58-66, 1999.

Evaluation of the influence of the corrosive media onthe corrosion resistance of 316L steel

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Stainless steels are widely used in the industrial environment due to their special properties such as mechanical and corrosion resistance. The 316L stainless steel has been used in the manufacture of the equipments for the chemical, petrochemical and pharmaceutical industries [1-2]. The corrosion resistance of stainless steels is mainly due to the presence of the oxide film spontaneously formed on the metallic surface and that provides protection by passivation. However, in the presence of an aggressive environment, such as chloride ions presence, in addition to other factors such as temperature and pH of the corrosive medium, these materials can become susceptible to different types of corrosive processes [2-3]. Considering the costs of the corrosion and the wide range of applications for 316L steel, this work evaluated the effect of different environmental conditions on the susceptibility to corrosion of 316L steel. The steel samples were analyzed in three corrosive media that simulate different aggressiveness: 3.5% NaCl, 3.5% $Na_2SO_4$and 3.5% HCl. The samples were exposed to the medium at two different temperatures: 25° and 70° C. The corrosion resistance was evaluated through electrochemical and immersion tests. The results showed that the 316L steel showed lower corrosion resistance in the presence of chloride, especially at acid medium. In tests at room temperature, the presence of pitting corrosion was observed. Immersion tests at a temperature of 70 ℃ showed the presence of uniform corrosion in addition to pitting. In sulfate medium, no significant corrosive processes were observed on the surface of the sample.

Acknowledgements:
FAPERJ and UERJ for finantial suport

References:
[1] OSTOVAN F, et al, Journalof materials research and technology, 2021 (13), p 2115-2125.
[2] STOYCHEV D, et al. Materials Chemistry and physics, 2022 (73), p 252-258.
[3] TIAN H, et al. Journal of Electroanalytical Chemistry, 2021(894), p. 115368

EVALUATION OF THE USE OF EDTA FOR EXTRACTION OF POTENTIALLY TOXIC METALS FROM PCB WASTE

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Waste from Electrical and Electronic Equipment (WEEE) has been raising increasing environmental concerns due to the rapid technological evolution and the growth of electronic consumption. These wastes may contain high-value metals, such as gold, silver, palladium, and rare earth metals, as well as potentially toxic metals, such as lead, cadmium, mercury, and arsenic, which are harmful to the environment and human health. In this context, hydrometallurgical techniques for recovering these metals can be employed in the minimization and remediation of environmental problems associated with metals. Thus, this study investigated the efficacy of ethylenediaminetetraacetic acid (EDTA) in removing potentially toxic metals, such as copper, lead, zinc, nickel, and aluminum from milled wastes of printed circuit boards (PCBs) from computers. The initial mass of metals in the waste was determined through digestion of the milled PCBs in aqua regia, followed by an analysis using inductively coupled plasma optical emission spectrometry (ICP-OES). To extract the mentioned metals, EDTA was used at a concentration of 0.6 M and a temperature of 80°C. The results demonstrated that EDTA has the potential to recover metals from PCBs, showing an extraction content of up to 54.1%, 33.1%, and 44.0% for aluminum, lead, andzinc, respectively. The application of EDTA may contribute to sustainable practices of hydrometallurgical techniques employed in electronic recycling, as well as reducing the cost of reagents and the environmental impact caused by the disposal of effluents generated using other leaching agents.

Evolution of Residual Stress on AISI D2 tool steel short time tempering

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Some parameters are used in an attempt to estimate a relationship between time and tempering heat treatment temperature of steels, among which the Hollomon-Jaffe parameter stands out [1]. This work aimed to study the evolution of residual stresses in short-time tempering treatment of AISI D2 tool steel, compared to conventional heat treatment of tempering [2]. The samples were treated in dilatometry for a better control of the thermal processing variables. Microstructural images were generated using optical and scanning electron microscopy, including mapping using EDS. Vickers microhardness measurements were performed. And the measurements of variations in residual stresses in each of the treatment stages and at the end of the treatment cycles were performed by analyzing the X-ray diffraction data using the Sen2Ψ technique [3]. The cycles composed of tempering in short times carried out at 500 ºC or 600 ºC, for 10 seconds or 1 minute, exhibited residual stress states similar to the conventional cycle, with a tendency towards neutrality of the residual stress state and high hardness values. The final residual stresses acquired by the AISI D2 tool steel in the thermal cycles studied, more specifically in the tempering, can be attributed to the precipitation of tempering carbides, which when carried out up to 600 ºC, occurs coherently, or at least partially coherently, to the matrix, producing the neutrality of residual stresses, but when tempering is carried out at 700 ºC, it leads to incoherent precipitation of carbides and matrix recrystallization, leading to an increase in compressive residual stresses and a reduction in the hardness of the material, regardless of the heat treatment time of tempering.

Acknowledgments:
Sponsorship agency CAPES.

References:
[1] J.H.Hollomon and L.D.Jaffe, Transactions of the AIME, v.162,p.223(1945)
[2] N.Schmidtseifer And S.Weber, Metallurgical and Mat. Trans. A, v.172,p.511(2011)
[3] J.Johansson et al., Acta Materialia, v.47,p.2669(1999).

Experimental investigation of phase equilibria at 1100 °C in the Cr-Nb-V system

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Currently, a new class of metallic alloys, known as high entropy alloys (HEAs) or multi-principal element alloys (MEAs), is being investigated by the scientific community. These alloys combine five or more elements in equiatomic or semi-equiatomic proportions. Aiming at structural applications at high temperatures, such as in the aerospace industry, metals with high melting point and relatively low density are important constituents of these alloys. In this context, the Cr-Nb-V system, object of the present study, is a key ternary to develop multi-principal element alloys for high temperature applications. As little information is available for this system, an isothermal section at 1100 °C is proposed for the first time in the literature based on the results of experimental investigation in this work. This proposal is based on the microstructural characterization of 25 alloys, which were arc-melted under argon, heat-treated at 1100 °C for 20 days, carried out by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and X-ray diffractometry (XRD). The results showed that the maximum solubility of vanadium in the C15 Laves phase is approximately 34 at.%, which is compatible with the increase in vanadium solubility with decreasing temperature indicated in isothermal sections at different temperatures (1350 and 1000 ºC) published in the literature [1,2].

Acknowledgements:
The authors gratefully acknowledge the financial support from the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brazil (CAPES) - (contract number 88887.604069/2021-00)

References:
[1] TAKASUGI, T.; YOSHIDA, M. TAKASUGI, T.; YOSHIDA, M.; HANADA, S. Journal of Materials Research, v. 10, p. 2463-2470, 1995.

[2] FEDOTOV, A.K. Izvestiya Akademii Nauk SSSR, Metally, v. 4, p. 207-212, 1974.

FAILURE ANALYSIS OF THE REAR TIP OF ASUGARCANE LOADER

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This work deals with the failure analysis of rear differential shaft of a sugar cane loader that broke in a little less than three years of service. For the analysis of fracture, were realized fracture surface macrographs, and microhardness profile metallographic analysis. Was also carried out numerical simulation of shaft estimating reliable way external efforts suffered during use. For this numerical simulation was performed in linear elastic regime. The microstructure is in agreement with the manufacturer. The surface hardness was 60HRC according to which the manufacturer presented. The failure occurred by high cycle fatigue and we observe various nucleation points to a singularity near the radius of curvature of the shaft. The numerical simulation was possible to quantify the tensions and showed us that this singularity in the axle base was what caused tension concentrations that result in the failure of the component.

Generalized Quasichemical Approximation applied to superconducting materials

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The search for new, efficient superconductors has been a central focus of research in solid-state physics and chemistry for some time. To predict the behavior of these materials, it is crucial to develop efficient models for their thermodynamics. In this study, we introduce an ab-initiostatistical model called the Extended Generalized Quasichemical Approximation (EGQCA) to investigate off-stoichiometric superconductors. Using EGQCA, one can predict physical properties, such as the critical temperature of any alloy as a function of composition and crystal growth temperature. The model evaluates the average of individual supercell properties weighted by their occurrence probabilities to minimize the overall mixing Gibbs free energy. We demonstrate the effectiveness of EGQCA by applying it to Al-doped MgB2, which shows excellent agreement with experimental data. Our open-access and user-friendly implementation of EGQCA makes it applicable to any 2D or 3D system, and enables high-throughput screening of complex high-Tcsuperconductors, providing insights into the interplay between synthesis and thermodynamics in these materials.

The project was financed by grant #2020/08258-0, São Paulo Research Foundation (FAPESP).

Graded NiAl/WC coatings

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NiAl intermetallic coatings have been used to protect superalloys from high-temperature operating conditions [1]. When processed in-situ through Directed Energy Deposition-Plasma Transferred Arc (DED-PTA), on AISI 304 austenitic stainless steel, high levels of dilution with the substrate are measured due to specific features such as the exothermal synthesis of β-NiAl phase [1]. This leads to the presence of large amounts of austenitic γ-FeNi phase, together with the aluminides. Increasing amounts of the austenitic phase, which has lower hardness, results in a decrease in the overall coating hardness, affecting the tribological properties [2]. The amount of γ-FeNi phase can be reduced by depositing multi-layers [3]. Furthermore, multi-layers can be processed offering gradual changes in composition, allowing to manufacture Functionally Graded Materials (FGMs). In this work, FGMs were processed by DED-PTA using Ni and Al powder mixtures with a gradual increase in WC content. Results show a reduction in the effects of the austenitic phase in the aluminide coatings. Also, the gradual increase of WC fraction in the powder mixtures induces multilayers with a graded functionality taking advantage of the microstructural changes and improvements in hardness and wear that a distribution of carbides provide.

Acknowledgments:

CAPES and LAMES Laboratory (UFPR).

References:

[1] Noebe R.D., Bowman R.R., Nathal M.V. (1996). The Physical and Mechanical Metallurgy of NiAl. In: Stoloff N.S., Sikka V. K. (Eds.) Physical Metallurgy and processing of Intermetallic Compounds. Chapman Hall, New York, pp. 212-296.
[2] Abreu-Castillo, H. O., Bueno, B. P., D' Oliveira, A. S. (2022). In situ processing aluminide coatings with and without tungsten carbide. The International Journal of Advanced Manufacturing Technology, 119(1), 477-488.
[3] Mazur, V. T., Mazur, M. M., D''Oliveira, A. S. C. (2022). Graded Inconel 625 coatings with in-situ processing of Ni3Al. Surface and Coating Technology, 445, 128660.

Green synthesis and characterization of metallic nanoparticles prepared using Solanum Lycopersicum extract (tomato)

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The emerging Green Chemistry area is the use of principles that reduce or eliminate the use or generation of hazardous substances in the design, manufacture and application of chemicals and envisages harm reduction as a performance criterion when designing new chemical processes. Green chemistry approaches to nanoparticle synthesis are one-step, fast, simple, low-cost, avoid toxic chemicals, and use non-toxic solvents such as water. Therefore, the use of plant extracts as a reducing agent in the synthesis of nanoparticles hasreceived increasing interest. Being easily reproducible and biocompatible, together with the availability of simple protocols, make this approach a favorable procedure for nanoparticle synthesis. In this context, this study aims to develop two types of metallic nanoparticles, based on Sn and Nb, from the use of Lycopersicon esculentum extract (tomato) through a route considered green [1,2]. These nanoparticles have the potential to be applied to capture gases and medical field. Afterwards, these nanoparticles will be characterized by UV-Visible spectroscopy, Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) measurements and scanning electron microscopy (SEM). The results showed that nanoparticles are promising for use as catalysts in gas capture systems.

Acknowledgements:
We gratefully acknowledge financial support from Fundac¸a˜o de Amparo a` Pesquisa doEstado do Rio de Janeiro (FAPERJ) (Ref. Proc. E-26/211.435/2019).

[1] H.E. Garrafa-Galvez et al. Green synthesis of SnO2 nanoparticle using Lycopersicon esculentum peel extract. Journal of Molecular Structure 1197, 354e360, 2019.

[2] Gómez-López et al. Nanomaterials and catalysis for green chemistry. Catalysis for green chemistry, Current Opinion in Green and Sustainable Chemistry, 24:48-55, 2020.

High-Resolution Chemical Screening of a Cr-Co-NiMulti-Principal Element Alloy Processed via High Pressure Torsion

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In recent years the multi-principal element alloy (MPEA) design approach has gained a significant attention from the scientific community due to its vast range of possibilities. Face centered cubic (FCC) MPEAs, have demonstrated their potential for multiple engineering applications, including the Cr-Co-Ni alloy, which is one of the toughest materials known. This alloy's exceptional properties are attributed to the plasticity-induced deformation mechanisms, including twinning and phase transformation. However, there is still much that remains elusive in the understanding of the deformation and structural behaviors of MPEAs. Studies show that the slight local preference between the elements can lead to chemical fluctuations and even local chemical ordering (LCO) [1,2]. The stacking fault energy is highly dependent on the chemical composition, and thus, the deformation mechanisms can be impacted by these fluctuations [1,2]. To further investigate this, we manufactured a $Cr_{40}Co_{30}Ni_{30}$ alloy through arc-melting of the high purity individual elements. The material was thermo-mechanically processed multiple times with cold rolling followed by annealing. Then, the fully recrystallized sample was severely deformed via high pressure torsion (HPT). To analyze the material, we removed a thin foil for analysis from the near-border region of the disk using a focused-ion beam setup coupled on a scanning electron microscope. This foil was characterized in a transmission electron microscope with high-resolution energy-dispersive X-ray spectroscopy. Local chemical fluctuations were detected in both the sub-micrometer and atomic scales.

Acknowledgments:
This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - 88887.621834/2021-00. We also acknowledge the LME/LNNano/CNPEM for the TEM characterizations.

References:

[1] J.Ding et al., PNAS vol. 115, pages 8919 (2018).
[2] L.Zhou et al., Acta Materialia vol. 224, page 117490 (2022).

High temperature oxidation behavior of Ni based superalloys derived from RR1000 alloy through Alloys-By-Design protocol

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A new class of Ni-based superalloys was recently developed by optimizing the RR1000 alloy [1]. The chemistry of these alloys, designated by ABD-2, ABD-4 and ABD -6, differ by the partial replacement Ti by Nb. [2] studied the oxidation of these alloys at 800°C for 100 hand noticed an enhancement of the resistance with the increase of the Nb content. In thiswork, the effect of the Nb and Ti on the oxidation resistance was investigated for two alloys:
i) ABD-Ti having the same chemistry of the ABD-2 from [1], containing only Ti, and ii) ABD-Nb containing only Nb. The alloys were cast by arc-melting under Ar, solubilized at 1170°Cfor 2 h and aged at 850°C for 4 h. The oxidation tests were carried in isothermal andpseudo-isothermal conditions at 800 and 900°C. At both temperatures, the ABD-Ti alloy presented a parabolic kinetic with rate constants of $4.3E^{-7}$and $9.1E^{-6}mg^{2}.cm^{-4}.s^{-1}$at 800 and 900°C respectively. At 800°C, the ABD-Nb alloy showed a parabolic behavior at the beginning of the exposure up to 280 h. Near 300 h a kinetic transition takes place with astagnation of the mass change up to the end of the test. At 900°C the ABD-Nb followed aparabolic behavior with a rate constant of $1.7E^{-6}mg^{2}.cm^{-4}.s^{-1}$. For both alloys, the characterization by XRD indicated the presence of Cr_2O_3, $Al_2O_3$and $(Ni,Co)Cr_2O_4$for the specimens tested at 800 and 900°C. These results are corroborated by cross section micrographs that also showed the presence of internal Al_2O_3. For the ABD-Nb alloy oxidized at 800°C, XRD and SEM characterization showed the presence the δ phase in the subsurface region that can be formed by means of oxidation induced transformation and the formation a continuous Al_2O_3layer enhancing the oxidation resistance of the alloy.

[1] CRUDDEN, D. J.; et al., Metall. Mat. Trans. A, 44 (2013) 2418
[2] LAPINGTON, M. T.; et al., Acta Materialia, 206 (2021) 116626

Hydrogenation reactions of the Ti31,8V33,7Nb29,8Co4,7 multicomponent alloy

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Hydrogen is often suggested as an ideal substitute to fossil fuels in energy carrier applications. Developing more efficient and safe hydrogen storage systems might be the key factor to facilitate its applications. Solid state storage via metal hydrides is a suitable option, especially considering stationary applications. Multicomponent body-centered-cubic (BCC) alloys have been showing promising storage properties. However, the comprehension of the absorption/desorption reactions of these alloys is still in the early stages. In this work, we investigated the hydrogenation reactions of the Ti31,8V33,7Nb29,8Co4,7 multicomponent alloy. The alloy was produced via arc melting and it was characterized via Scanning Electron Microscopy (SEM) and X-Ray diffraction (XRD). The alloy was hydrogenated with different concentrations of hydrogen and subsequently characterized via XRD. Desorption reactions were analyzed via Thermal Desorption Spectroscopy (TDS) of the hydrogenated samples. The as-cast Ti31,8V33,7Nb29,8Co4,7 alloy presented a BCC structure with dendritic morphology. The XRD and TDS analyses of partially and fully hydrogenated samples indicate that the alloy presents the following hydrogen absorption/desorption reactions: BCC alloy ↔ BCC intermediate hydride ↔ FCC dihydride. Similar sequence of reactions has been reported for the (TiVNb)85Cr15 alloy in the work developed by Silva et al. [1]. However, in this work, the coexistence of two BCC phases at the intermediate level of hydrogenation was identified using only conventional laboratory XRD analyses.

Acknowledgements:

Serrapilheira Institute (Serra-1709-17362), FAPESP (2020/07707-6), CNPq (Grant number: 309467/2021-7 and 407906/2022-3). This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001, Federal University of Sao Carlos, Graduate Program in Materials Science and Engineering.

References:

[1] B. H. Silva et al., JALCOM 901, 163620 (2022)

Hydrogen storage properties of the equiatomic TiNbCr alloy

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Meeting global energy demands while minimizing environmental impact is an ongoing challenge. One promising solution is the use of hydrogen as an energy vector, which has a high gravimetric energy density and produces only water as a byproduct. However, safe and efficient hydrogen storage remains a critical barrier to its widespread use. Solid-state hydrogen storage through metal hydrides is a promising option and Ti-Nb-Cr is one of many alloy systems currently being researched, although only a few studies have explored its properties in depth. Therefore, this study aims to characterize and investigate the hydrogen storage properties of the equiatomic TiNbCr alloy. The alloy was produced by arc melting and a X-ray diffraction analysis revealed a body-centered cubic phase with a fraction of C15 Laves phase. The alloy absorbed hydrogen without prior heat treatment when exposed to 30 bar of H2 at room temperature, showing a maximum capacity of 2.80 wt% after an incubation time of 1 hour and 5 min of absorption. The application of heat treatment at 400°C for 2 hours under dynamic vacuum resulted in the absorption under 2 min with no incubation time. The XRD pattern obtained after the first hydrogenation at room temperature revealed a face-centered cubic phase along with the primary C15 laves phase. Additionally, the Pressure-Composition-Temperature (PCT) diagram was measured at temperatures ranging from 25°C to 150°C, showing a low plateau pressure of approximately 0.01 bar at room temperature.

Acknowledgements:
This study was supported in part by the grant n° 2022/06719-6 from São Paulo Research Foundation (FAPESP), in part by the Brazilian National Council for Scientific and Technological Development - CNPq (Grant number: 309467/2021-7 and 407906/2022-3) and in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001, Federal University of Sao Carlos, Graduate Program in Materials Science and Engineering.

Impact of Cryogenic Equal Channel Angular Pressing on the Microstructure and Mechanical properties of a Cu0.7Cr0.07Zr Alloy

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Equal Channel Angular Pressing (ECAP) at cryogenic temperatures has been adopted to increase the strain severity by suppressing dynamic recovery and accumulating crystalline defects. In this context, this study performed ECAP on a Cu0.7Cr0.07Zr alloy at room and cryogenic temperatures (-80°C) and analyzed its impact on microstructure and mechanical properties. Uniaxial tensile tests and Vickers hardness measurements assessed mechanical properties, while microstructural changes were evaluated using optical microscopy (OM), transmission electron microscopy (TEM), electron backscatter diffraction (EBSD), X-ray diffraction (XRD) and differential scanning calorimetry (DSC) analysis. The results showed that the material presented deformation twins after processing at both temperatures. Also, it was observed a decrease in grain size of around 94% and 88% after ECAP at room and -80°C, respectively, leading to a corresponding increase in hardness of 90% and 119%. Yield Strength also increased by 358% and 373% after ECAP at room and -80°C, respectively. However, these mechanical property improvements were accompanied by a reduction in the uniform elongation of 92% and 94% after ECAP at room and -80°C, respectively. Deformation twins was also observed after ECAP at both temperatures. Hence, the study highlights the effectiveness of ECAP at cryogenic temperatures in enhancing the mechanical properties of the Cu0.7Cr0.07Zr alloy.

Acknowledgements:
The authors would like to express their gratitude to Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES - process nº 88882.332697/2010-01)

References:

[1] T. Konkova, S. Mironov, A. Korznikov, S.L. Semiatin, Microstructure instability in cryogenically deformed copper, Scripta Materialia, 63 (2010) 921-924.
[2] T. Sakai, A. Belyakov, R. Kaibyshev, H. Miura, J.J. Jonas, Dynamic and post-dynamic recrystallization under hot, cold and severe plastic deformation conditions, Progress in Materials Science, 60 (2014) 130-207.

INFLUENCE OF HEAT TREATMENT OF QUENCHINGAND TEMPERING ON H11 MOD TOOL STEEL

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The heat treatment is a process that acts as a key factor in the tool life of tools made with hot work tool steels. Due to their applications, the main desirable metallurgical properties of this material class are toughness and hot resistance, without which the tools may suffer premature failure, and may lead to increases in the cost of producing articles made using such tools [1, 2]. Therefore, special attention must be given to the heat treatments practiced in these steels, whose operations involve quenching and tempering. The present work carried out the effects of the austenitizing and tempering temperatures on the microstructural condition, hardness and impact properties of modified AISI H11 with lower silicon content. Hardness was measured by the Rockwell method, C scale; the toughness was evaluated by the Charpy-V impact test; and the microstructure for each condition was evaluated by optic microscopy. It was shown that higher austenitization temperatures resulted in increased grain and hardness; higher tempering temperatures resulted in reduction of hardness, and secondary hardening peaks were observed for some austenitization conditions. The impact resistance showed an inverse behavior to that of the hardness, remaining initially constant, up to ~ 550 ° C of tempering temperature, and then increasing with the tempering temperature. The microstructures presented great dissimilarity, mainly due to the dissolution of carbon and alloying elements in the matrix. Another factor that impacts the final toughness of the material is the cooling rate during quenching; the higher it is, the greater the predominance of martensitic microstructure, obtaining greater resistance to impact. In the end it is possible to find the heat treatment that can balance good mechanical strength and good toughness.

Influence of light on the corrosive behavior ofcommercial alloys A356 and A413 used in the automotive wheels

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Lightweight materials with good mechanical properties are alternatives to reduce the mass and energy used in motor vehicles. Components cast with A356 and A413 alloys are used for this purpose. The production of these alloys, can improve the mechanical properties, but on the other hand, it can affect the corrosion resistance due to the formation of second phase. By inserting alloys into saline solutions, the corrosion develops, forming the corrosion products. Thus, this study aims to analyze the corrosion rate in for A356 and A413 alloys in the presence and absence of light (ASTM G1, 2003). A mixture alcohol: acetone (1:1, v/v) was used to drying. An analytical electronic balance (10^{-4}g) to obtain the weight values of the proposed alloys was used. In order to remove the corrosion products, all the samples passed through cleaning solutions (ASTM G1, 2003). After each one of the immersion periods by 6042 h, in order to evaluate the sample degradation (weight change, diameter and thickness), the samples were taken out and the cleaning was carried out. The pH analyses of corrosive media were also followed.Results show that samples (A413) with incidence of light suffered a mass reduction of ~42% (light: $3.1.10^{-6}$g/h; without light: $1.8.10^{-6}$g/h) in relation to those in the light absence. To understand the shape and corrosion products in alloys A356 and A413,the same behavior was not observed, but it was noted that after 3400 h, the Al particles became more defined in relation to A356. This may be related to the amount of Si that influenced the corrosive process due to the greater formation of the galvanic couple,in addition to the porosity of the material that can increase the surface area for NaCl to act.The pH results of the solutions show the most acidified medium n the light incidence(5.30) in relation to the absence of light (6.40).

References:

ASTM International. Standard practice for preparing,cleaning, and evaluating corrosion test specimens, ASTM G1-03, 2003.

Influence of phosphatization as a surface pre-treatment for electrodeposition nickel coatings on ASTM A36 steel substrates.

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The use of coatings is one of the most widespread techniques to combat the corrosion process, as it delays the access of the electrolyte to the substrate [1,2]. Nickel-based coatings have been a commonly used strategy, due to the anticorrosive properties, high hardness and good wear resistance of this type of material. Nickel plating obtained by electroplating is an advantageous option due to its fast execution and relatively low operational cost [3]. Phosphatization is a pretreatment that not only improves the adhesion of coatings to the substrate by forming a porous layer of inorganic precipitates, but also retards corrosion due to their non-conductive nature [1]. The objective of this work is to evaluate the influence of phosphatization as a pretreatment in the electrodeposition of nickel on ASTM A36 steel substrates. Four conditions were analyzed: uncoated steel (E00), phosphatized steel (E00F), nickel-coated steel (E10), and phosphatized and nickel-coated steel (E10F). Morphological and chemical characterizations were performed using SEM and EDS. The anticorrosive behavior was analyzed using EIS and potentiodynamic polarization. Wear resistance was analyzed by means of a tribometer. The phosphatization process influenced the resulting morphologies, leading to an increase in pointed lamellar structures on the surface, as well as an increase in anticorrosive protection and wear resistance, with the bests results observed in the phosphatized and nickel-coated steel sample.

Acknowledgments: FINEP, CAPES, CNPq, FACEPE PROPESQI-UFPE, LBC-COMPOLAB/UFPE, LITPEG/UFPE and INTM/UFPE.

References:

[1] Pacheco,W.F. and Semaan, F.S., Revista Virtual de Química 5, 516-537 (2013).

[2] Borkar, T. and Harimkar, S.P., Surfaces Coatings Technology 205,4124-4134 (2011).

[3] Darband, G.B. and Aliofkhazraei, M., Journal of Alloys and Compounds 818, 152843 (2020).

Influence of reduced graphene oxide on the fabricationto Neodymium-Iron- Boron magnets by mechanical milling

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The aim of this work was to prepare sintered Nd-Fe-B permanent magnets, following the traditional powder metallurgy route, using reduced graphene oxide (rGO) in the mechanical milling process. Also, it was to study the effect of rGO addition on the magnetic properties of the as-prepared materials. rGO acts as a lubricant and effective milling medium, preventing agglomeration, welding of the particles, and oxidation of the material. The structure and morphology of the composite powders were characterized by X-ray diffraction, Raman spectroscopy, and transmission electron microscopy. The magnetic properties of sintered magnets were characterized using a permeameter. The highest values of remanence (Br) and intrinsic coercivity (iHc) values were obtained for the sintered magnet using 0.02 wt% rGO addition, Br = 1.1 T, and iHc = 764.0 kAm-1

Influences of the Warm-Rolling Process in the Microstructure and Mechanical Properties of the CrMnFeCoNi High Entropy Alloy

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The influence of warm-rolling on the microstructure and mechanical properties of the FCC equiatomic CrMnFeCoNi high entropy alloy (HEA) was investigated. The HEA was warm-rolled to 76% and 90% reduction in thickness at 773 K. The warm-rolled HEA resulted in a lamellar microstructure, notably coarser than the cold-rolled material found in literature [1,2]. The difference in microstructure could be a consequence of the dynamic annihilationof dislocations during deformation. In addition, nano-twins were introduced during warm-rolling process. The chemical fluctuation in the HEA warm-rolled indicated the formation of second phases. The HEA warm-rolled in different thickness reductions resulted in a great combination of strength-ductility in which the high strength is due to a reduction in the free path of dislocation by nano-twins inside the grains and the ductility attributed to the recovery process during deformation. In summary, the present results indicate that the warm-rolling could be effectively used as a thermo-mechanical route for tailoring microstructure and mechanical properties of the CrMnFeCoNi alloy and possibly in other single-phase FCC HEAs.

Acknowledgements:
This work is supported by National Council for Scientific and Technological Development (grant number 423833/2018-9). The authors would like to acknowledge the Electron Microscope Unit of the University of New South Wales for providing the equipment and technical support for electron microscopy experiments. Fapesp Proc. 2021/04302-8

References:
[1] J. Gu, M. Song, Scr. Mater. 162 345-349 (2019).
[2] G.H. Sousa, E.M. Mazzer, Mater. Sci. Eng. A 793 (2020)

Inter and intragranular precipitation mechanisms of α phase in β metastable Ti alloy of Ti-Nb-Fe-Sn system.

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Beta metastable Ti alloys are potentially interesting as structural biomaterials. When it is solution heat-treated above the β-transus temperature and rapidly cooled, it is possible to obtain microstructures constituted by the β phase (bcc) with a low elastic modulus, which is essential to avoid the stress-shielding phenomenon. When aged, it can present the α phase (hcp) precipitation into a β phase matrix, which increases the mechanical strength of this material. By controlling the aging process, it is feasible to obtain components with low elastic modulus and high mechanical strength. For example, it is possible to adjust the elastic modulus to values between 60 GPa and 110 GPa and yield strength from 500 to 1000 MPa. When this precipitation is based on heterogeneous nucleation assisted by the ω phase, the precipitation is intragranular. However, aging can also produce intergranular precipitation assisted by grain boundaries. In this context, the objective of this work was to investigate the aging heat treatment conditions of the Ti-19Nb-2.5Fe-6Sn alloy (wt%) and explore the competition between intragranular and intergranular precipitation. The α phase precipitation was investigated through differential scanning calorimetry, X-ray diffraction, and scanning electron microscopy. The effect of intergranular/intergranular precipitation on mechanical behavior was evaluated. The results showed that the aging temperatures are essential in obtaining intragranular precipitation of the fine and dispersed α phase in the β phase matrix. Acknowledgments: We acknowledge the São Paulo Research Foundation, FAPESP, grant # 2018/18293-8 and# 2021/06156-9.

Investigation of the Effects of In on the SolidificationPath, Microstructure, and Interfacial Features of Sn-Bi- In Alloys for Lead-Free Soldering Applications

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Due to the high toxicity of lead, the electronic components industry has developed new lead-free alloys, which has become a topic of great scientific and technological interest [1]. The Sn-Bi alloys are considered good candidates in order to replace Sn-Pb alloys in soldering operations [2]. However, either the mechanical properties, or the control growth of the interfacial reaction layer must be improved. The addition of In has shown good results for applications in electronic connections using low melting point alloys based on Sn [3]. In this work the influence of In on the solidification path, microstructure, contact angle, tensile properties, and interfacial features of a Sn-40%Bi alloy was studied. CALPHAD calculations, differential scanning calorimetry, scanning electron microscopy, x-ray diffraction, wettability and tensile testing were performed. The microstructures were composed of Sn-rich matrices with Sn-Bi and BiIn-Bi constituents. Significant sensitivity of the microstructural scale to the solidification cooling rate was observed by employing different processing conditions. Because of the relatively high BiIn fraction on the microstructure, a limited elongation of approximately 7% was achieved, with an ultimate tensile strength of 65 MPa. The Cu6(Sn, In)5 intermetallic phase formed on the reaction film for the Sn-Bi-In/Cu interface under all tested conditions, including as-soldered and aged at 100°C and 120°C for 120 h, 240 h, and 360 h. Wetting angles for the ternary Sn-Bi-In alloy were maintained at approximately 20°, which is smaller than usual for the binary Sn-Bi alloys. The new phase, Cu6(Sn, In)5, produced instead of Cu6Sn5, showed a lower growth ratethan that observed in In-free couples.

Low-cost metallic glasses with high performance in corrosion and wear resistance from ferrous alloys and commercial elements.

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Metallic glasses offer a unique combination of properties, including high mechanical strength, corrosion resistance, and wear resistance [1,2]. However, research on alloys containing a glass phase is mostly carried out using high-purity precursors, which are costly. Therefore, there is an interest in developing metallic glasses from more accessible raw materials, such as ferrous alloys. This study aims to develop a high-corrosion- and wear-resistant metallic glass from pig iron, a low-value-added ferrous alloy with a high impurity content. Initially, a pig iron-based alloy with commercial purity elements such as Cr, Fe-B, Fe-Nb, and Fe-Mo was produced in an electric arc furnace and then processed through rapid solidification by melt-spinning. Subsequently, the obtained alloy was characterized using X-ray diffraction (XRD), differential scanning calorimetry (DSC), and transmission electron microscopy (TEM) to evaluate the formation of the glass phase, as well as electrochemical analysis. The results showed the formation of a glass ribbon with excellent corrosion and wear resistance. These findings are significant for the development of low-cost metallic glasses, which have a broad range of applications in advanced technology.

Acknowledgments:
This study was partially funded by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001. This work was supported by CNPq, Conselho Nacional de Desenvolvimento Científico e Tecnológico - Brazil - Finance code 161113/2021-4.

References:
[1] Inoue et al., Nature Materials vol. 2, pages 661-663 (2003)
[2] C Suryanarayana A Inoue., International Materials Reviews vol. 58, pages 131-166 (2013)

Machinability analysis of Al-4.5%Cu-1%Mg alloy: correlation between cutting temperature and solidification microstructure

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Modern industry has opted for the use of aluminum alloys in various engineering components due to its low-density combining lightness and high strength property. The aerospace and automotive sectors have therefore sought the machining process of these alloys as a more favorable finishing operation to obtain better dimensional accuracy and ideal surface finish in the field of parts manufacturing [1]. The removal of a material through contact between the cutting tool and the part generates a large expenditure of energy in the form of heat due to friction and plastic deformation [2]. Several tests can be carried out to assess the quality of the machining process, such as necking tests on the lathe with a cutting tool. The evaluation of these cutting temperatures allows them to be later controlled by knowing the cutting conditions (tool material, process parameters and environment) and even the alloy preparation process (with the control of thermal and microstructural parameters of solidification), promoting analysis of the impact that this heat can generate on tool life and wear mechanisms [3]. Hence, this work aims to study the cutting temperature obtained through the infrared method, during the necking process of the ternary Al-4.5%Cu-1%Mg solidified in a horizontal device. The results show a variable behavior of the heating rate during machining along the ingot. In the transition zone from columnar to equiaxed grains, there was a sharp drop in cutting temperature, suggesting that this zone has a lower hardness value.

[1] T. R. Soren, R. Kumar, I. Panigrahi, A. K. Sahoo, A. Panda, and R. K. Das. Materials Today: Proceedings 18, 5069-5075 (2019).

[2] K. Mahesh, J. T. Philip, S. N. Joshi, and B. Kuriachen. Materials and Manufacturing Processes 36, 753-791 (2021).

[3] S. L. de P. Silva, J. M. S. Dias Filho, C. F. dos Santos, T. A. P. de S. Costa, O. F. L. da Rocha and M. A. P. de S. da. Silva. Brazilian Journal of Development 7, 60959-60971 (2021).

Magnetic domain characterization of Fe-Ga and Fe-Ga-B alloys

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Low hysteresis and moderate magnetostriction at low magnetic fields of FeGa alloys makes it attractive for actuator and sensing applications. The alloy with better magnetostriction contains 18.6at.% of Ga, but shows poor ductility. The addition of B on these alloys improves ductility without affecting magnetostriction. Magnetostrictive strain comes from magnetization rotation and thus is closely related to its magnetic domain patterns. The configuration and evolution of magnetic domain structure under a magnetic field are important for understanding the magnetostrictive response of materials. Magnetic force microscopy was used for the investigation of the magnetic domains of FeGa alloys. Recently was reported a significant development in FeGa alloys magnetic domain, where cellular domain structures were observed [1]. The magnetic domains of FeGa alloys with added B has not yet been explored. In this work, the magnetic domains of $Fe_{81.4}Ga_{18.6}$ and $(Fe_{81.4}Ga_{18.6})_{0.98}B_2$fabricated by arc furnace melting with Ar-controlled atmosphere are analyzed. The magnetic properties of these alloys were previously reported [2]. For the analysis, the microstructural characterization of the samples is performed using a scanning electron microscopy. The local evolution of magnetic domain structures in FeGa e FeGaB under variable magnetic fields (0-300 mT) is characterized via magnetic force microscopy and magneto-optical Kerr microscopy.

Acknowledgments:

The authors thank Brazilian scientific funding agencies CAPES and CNPq. Dr. Cristina B. Nunes thanks FAPESP for the financial support (project number 11/21258-0). Dr. Felipe Alencastro thanks FAPERJ for the financial support (project number 260003/019684/2022).

References:

[1] M.N. Tianen et al. Applied. Physic. Letter 117 (2020)

[2] C. Bormio-Nunes and F.M. Cardoso. Journal of Materials Research 33, 2207-2213 (2018)

Magnetic response of cobalt oxide ultra thin films

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Cobalt thin films were evaporated in a high vacuum system on mica and SiO2 substrates. The film thicknesses were varied between 1 and 15 nm. The samples were oxidized at RT, 100ºC, 150ºC and 200ºC, at ambient conditions by 1 hours. Raman spectroscopy and ellipsometry were performed in all samples. The characteristic Raman shift peaks corresponding to CoO (200, 480 and 690 cm-1) appear clearly in samples oxidized at RT and 100 ºC; they can be weakly identified in those oxidized at 150ºC; and they disappear in thefilms oxidized at 200 ºC. On the other hand, the values obtained from the ellipsometry wereconsistent with a refractive index of CoO. The magnetic characterization of the samples wasperformed, determining the hysteresis loop of all samples. Results shows no ferromagneticresponse from 5 nm down. From 10 nm thick to up, ferromagnetic signal is observed. Thebehavior is maximized for samples with 10 nm thickness, reaching a saturation field of 1E-7Oe and magnetic coercivity of 33, these values increase as the oxidation temperature and thickness increase.

Acknowledgement

This research was partially supported by ANID Fondecyt 1231849.

Methodology for developing a model to predict CCT diagrams of O&G steels

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For production of high end seamless steel pipes, which are obtained by quenching and tempering treatments, the continuous cooling transformation (CCT) diagram is a very important tool to comprehend the phase transformation and to predict the final microstructure of the steels. However, it is difficult to obtain these diagrams for every steel since a high experimental effort is necessary. This study presents a methodology of modelling using linear regression method to determine the relationship between the chemical composition of steels and the austenite transformation kinetics during the continuous cooling. The developed model makes possible to calculate the CCT diagram for steels with a known chemical composition and to analyze the influence of specific elements on the transformation curves of austenite. The methodology was developed first on the basis of information available in literature consisting of equations used for constructional steels and then it was adapted for Oil and Gas steels using experimental data obtained by dilatometry tests. The model presented in this paper is a useful tool in selection of the chemical composition of steels for heat treatment of pipes for O&G applications.

MICROSTRUCTURAL ANALYSIS AND THERMAL PARAMETER CHARACTERIZATION OF DIRECTED SOLIDIFICATION OF AL-3.2BI-0.5ZN ALLOY UNDER TRANSIENT HEAT EXTRACTION REGIME.

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Monotetic Al-based alloys present microstructural characteristics that make them notable for specific applications, such as in tribological systems. The binary system Al-Bi has been the subject of important investigations from the point of view of phase transformations and microstructural characterization. On the other hand, little is known about the effect caused on the microstructure - phases formed and morphology - by the addition of a new Al-Bi-X alloying element. In this investigation, 0.5% by mass of Zn was added to the monotetic alloy Al-3.2%Bi [1]. Considering that the added element has high solubility in the α-Al phase, it was expected that the microstructural morphology of the ternary alloy would remain the same as that of the binary monotetic. The alloy investigated in this work was directionally solidified in order to allow the obtainment of wide ranges of solidification thermal parameters and interphase spacings [2]. Optical micrographic analysis (OM) shows a uniform aspect matrix, without the presence of relevant boundaries, and globules with Bi presence, besides empty cavities, probably due to Bi detachment during grinding and/or polishing stages. The images recorded through the Scanning Electron Microscope (SEM) with Energy Dispersive System (EDS) module allowed confirming that the microstructure of the ternary alloy Al-3.2%Bi-0.5%Zn corresponds to the α-Al matrix, rich in Al and Zn, with planar morphology, and Bi globules dispersed along the primary phase.

References:

[1] SILVA, M.A.P.S.; SPINELLI, J.E.; GARCIA, A. Thermal parameters and microstructure during transient directional solidification of a monotectic Al-Bi alloy, Journal of Alloys and Compounds, Volume 475, Issues 1-2, 2009.

[2] COSTA, T.A.P.S.; DIAS FILHO, J.M.S.; FREITAS, E.S.; CASTELETTI, L.C., GARCIA, A. The effect of microstructure length scale on dry sliding wear behaviour of monotectic Al-Bi-Sn alloys, Journal of Alloys and Compounds, Volume 689, 2016.

Microstructural evaluation via EBSD/EDS techniques of the stainless steel 309L cladding welded by the MAG process

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Stainless steel 309L is used in the oil and gas industries to coat pipelines and equipment, standing out for having a low cost and good anti-corrosion properties, presenting in its composition high concentrations of chromium, nickel and low percentage of carbon, which increases weldability and the corrosion resistance of cladding components. For the production of the claddings, the MAG (Metal Active Gas) welding process was used and as a result, Partially Diluted Zones (PDZ) were formed - which present microstructural variations depending on the process parameters used - these, contributing and significantly influencing the final mechanical-metallurgical properties of the claddings [1]. In order to know and optimize the mechanical and anti-corrosion properties, it is extremely importantto know the microstructural aspects in order to understand the metallurgical transformations in the Bonding Zone (BZ) and, in this way, to investigate the influence of welding parameters on the formation of PDZs. With this, a microstructural evaluation of the dissimilar claddings was carried out via Electron Backscatter Diffraction (EBSD), with the objective of performing the microstructural mapping and understanding the influence of the welding parameters on the microstructure of these regions. In addition to the EBSD, the technique of Energy Dispersive Spectroscopy (EDS) and microhardness were used, in order to correlate the microstructural and chemical analyses with the mechanical properties in the cross-sectional region of the cladding, specifically in the PDZs.

Acknowledgements:
The Programa de Recursos Humanos da Agência Nacional do Petróleo, Gás Natural e Biocombustíveis - PRH-ANP and the Coordenação de Aperfeiçoamento de Pessoal de NívelSuperior - CAPES, for the financial support supported.

References:
[1] S. Wu, Z. Liu, X. Huang, Y. Wu, Y. Gong, International Journal of Refractory Metals and Hard Materials., 101 (2021).

Microstructural evolution of Al-Al3Ti-based alloy prepared by sintering of commercial powders

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The use of less expensive powders for the synthesis of materials results in a lower final product price, turning them more attractive for industrial applications. However, the quality of raw materials including impurities in their chemical composition, as well as the particle size distribution and morphology might influence the mechanical properties. In this context, the use of low-cost commercial-grade powders still requires investigation for the processing of Al-Al3Ti-based alloys. Therefore, this work aimed at evaluating the processability, microstructural evolution, and hardness of an Al-Al3Ti-based alloy prepared via Powder Metallurgy of commercial powders. Two powder mixtures with different powder particle sizes; both with approximately 26 wt.% of Ti; were uniaxially cold-pressed at 200 MPa, and sintered at 700°C for 30, 90, 120, and 240 minutes. In both cases, after 240 minutes of sintering, a homogenous microstructure was observed, with low porosity, and significantly refined and dispersed intermetallics.

Acknowledgment

The authors would like to thank the DEMM/FEM/UNICAMP and the São Paulo Research Foundation (FAPESP), Grant # 18/04564-0.

References

[1] Y. Ma, Q.S. Mei, C.L. Li, L. Wan, J.Y. Li, F. Chen, X.M. Mei, Z.H. Chen, Microstructure and mechanical behavior of Al-TiAl3 composites containing high content uniform dispersionof TiAl3 particles, Materials Science and Engineering: A, Volume 786, 2020, 139435.

[2] S. Vorotilo, A.A. Nepapushev, D.O. Moskovskikh, V.S. Buinevich, G.V. Trusov, D. Yu. Kovalev, A.O. Semenyuk, N.D. Stepanov, K. Vorotilo, A.Y. Nalivaiko, A.A. Gromov, Engineering of strong and hard in-situ Al-Al3Ti nanocomposite via high-energy ball milling and spark plasma sintering, Journal of Alloys and Compounds, Volume 895, Part 2, 2022, 162676.

[3] Siming Ma, Xiaoming Wang, Mechanical properties and fracture of in-situ Al3Ti particulate reinforced A356 composites, Materials Science and Engineering: A, Volume 754, 2019, Pages 46-56.

Microstructure and hardness of Al-3, -5, and -10wt.%Mg-0.1wt.%Sc alloys subjected to laser surface remelting

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Al-Mg-Sc alloys can be applied in different industrial sectors due to the relatively high specific strength and adequate resistance to corrosion. The purpose of this work is to process Al-3, -5 and -10wt.% Mg-0.1wt.% Sc alloys (commercially known as Scalmalloy) both by upward directional solidification (DS) and by laser surface remelting (LSR), and, after that, to evaluate the microstructures, phase morphologies and hardness. The two solidification techniques applied are complementary and allow not only the mapping of the microstructure as a function of thermal solidification parameters, but also the analysis of samples under extreme conditions of solidification when processed by LSR. LSR is an effective way to increase the hardness of Al alloys by refining the microstructure of the surface layer. Surface remelting was performed using an Yb:fiber laser under Argon protection. Four (4) laser heat inputs were examined for alloy substrates extracted from the DS samples and corresponding to the same solidification rate (2°C/s): 1.7, 2.5, 5.0 and 10 J/mm. Thermodynamic simulations (CALPHAD), optical microscopy, hardness, X-ray Diffraction and SEM analyses were employed to investigate the phases formed. The upward DS caused a microstructural and morphological variety in the three Al-Mg-Sc alloys with presence of cells and dendritic morphologies. Thermodynamic simulations indicated the formation of the $Mg_2Al_3$and Al_3Sc phases for the three DS compositions. For the lowest heat inputs (1.7 and 2.5 J/mm) Vickers hardness of the Al-3% and -5%Mg(-Sc) LSRed samples were increased approximately 15-20% as compared to the DS samples. However, in the case of the Al-10%Mg-0.1Sc alloy treated at 1.7 J/mm only a small hardness increase was observed from 114 HV (DS) to 118 HV (LSR).

Acknowledgements:

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001

Microstructure and hardness of warm and cold rolled Ti/Nb-IF steel

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Interstitial free (IF) steel is well-known by its excellent deep drawing performance. Variations in the thermomechanical processing can have strong effect on microstructure and mechanical properties of IF steels [1,2,3]. In order to investigate the effect of deformation temperature on microstructure and hardness of Ti/Nb-IF steel, two distinct deformation processes were carried out. The first strip (WR) was 80% warm rolled and the second strip (WCR) was 40% warm rolled and 40% cold rolled. Both strips were annealed at 900°C for 120 seconds. The annealed WR and WCR samples exhibited recrystallized microstructures with similar mean grain size values, 98 μm and 91 μm, respectively. According to these results, 40% cold deformation after warm deformation has shown not to be able to significantly increase dislocation density and deformation energy to promote nucleation of a higher number of new grains. The microhardnesses for the two annealled samples were also similar, about 83 HV. The texture results however, showed a positive effect of cold deformation in the previously warm rolled sample. After warm rolling, Goss texture component was developed but neither alfa nor gamma fiber were. On the other hand, warm rolling followed by cold rolling resulted in an intensification of the alfa fiber texture, which was beneficial for the further development of gamma fiber texture after annealing processing.

Acknowledgements:
The authors would like to acknowledge Companhia Siderúrgica Nacional, Instituto Militarde Engenharia and Centro Federal de Educação Tecnológica-CSF/RJ.

References:

[1] W.Chang, W.Yu, H.Yang, Z.Man and Y.Cao, Materials Science Forum, vol.993, 505 (2020)
[2] L.Li, Z.Cai, S.Wang, H.Wu, Y.Feng, Y.Liu and X.Mao, Metals, vol.11(9), 1341 (2021)
[3] A.Huang, Z.Wang, Q.Yuan, R.Chen, J.Qin and Y.Zhang, Metals, vol.12(7), 1178 (2022)

Microstructure and properties of Al-3, -5 and -10 wt. % Mg-0.1wt. % Sc alloys solidified at different cooling rates

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Aluminum alloys are very employed in engineering devices like those from aeronautical, aerospace industry and microelectronic, among others, due to combination of low density, mechanical strength and adequate corrosion resistance. It is well Known that the microstructure of these alloys depends on both the alloy composition and the processing conditions, so more in-depth studies regarding their solidification still need to be carried out. Then, the search for alloys with higher specific strength is justified from environmental and economic point of views. The aim of this work is to investigate the influence of cooling rates on the phase morphology, phase transformation and mechanical properties of three ternary Al-3, -5 and -10wt.% Mg-0.1wt.% Sc alloys, whose composition covers the spectrum of alloys known commercially as Scalmalloy. These alloys were processed by upward directional solidification whose a wide range of cooling rates results on the formation of a microstructural variety along the longitudinal cooling direction and hence different tensile properties.The upward directional solidification process caused a microstructural and morphological variety in the three Al-Mg-Sc alloys. Cells and dendritic morphologies were observed by optical microscopy. Thermodynamic simulations (CALPHAD Method) indicated formation of the beta phase intermetallic (Mg_2Al_3) and Al_3Sc phase for the three compositions. Superficial residual marks observed in the SEM analyses indicated indirectly the presence of Mg_2Al_3. Thermal analysis (DSC) confirmed the beginning features of solidification for each investigated alloy. The observed improvement in tensile properties can be associated with microstructural transformations and the mechanism of increased resistance by solid solution (Al-Mg) together with the formation of the Al_3Sc precipitates, whose detection is still a work in progress.

Acknowledgements:

This study was financed in part by the CAPES - Brasil - Finance Code 001

Milling effect in the Cu extraction process from printed circuit boards (PCB) in the acid-leaching solution

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Among the waste of electrical and electronic equipment (WEEE), the printed circuit boards (PCB) present the highest valuable metals' concentration - Au, Ag, Cu, and Sn. Such metalscan be recovered in a process known as urban mining [1]. In this scenario, the present work studied selective Cu extraction from microcomputer PCB from two different particle s by hydrometallurgical technique. The samples were ground and fractionated. They were divided into gross (0.71 to 2.84 mm) and fine fraction (0.71 mm). The Cu extraction was performed by leaching processes - 2 M H_2SO_4 added $H_2O_2$30% (p./v.), and the solid/liquid ratio was 1/10. Each leaching process was performed under magnetic stirring (200 rpm) for 1 h at room temperature (30°C). After the leaching process, the solution was filtered and stored. Successive washes with distilled water were also performed and the filtrate was stored. Both leach and wash water solutions were analyzed for the presence of Cu by inductively coupled plasma-optical emission spectroscopy (ICP-OES). The total Cu presentin the fine and gross samples was obtained by analyzing the final solution also by ICP-OESafter the acid digestion process (3/4 HCl and 1/4 HNO_3, both in analytical grade). The extraction percentage (EP) for fine and gross fractions reached (69±2) % and (44±1) %, respectively. As expected, the extraction of Cu in smaller particle fractions tended to increase the EP due to the enhanced surface contact area of the solid with the leach solution. Therefore, aiming for greater EP, it is recommended that prior milling to micronize the PCB.

Acknowledgements:

This work was supported by CAPES scholarship - Brazil (88887.680349/2022-00), FAPESB scholarship (BOL0273/2021), Catalisa ICT (Project 29083), and CNPq Universal (Project 421814/2021-7). The chemical characterization tests (ICP-OES) were kindly performed at LACOR-UFRGS.

References:

[1] PENG, P. and PARK, A.-H. A. Green Chemistry, v. 22, n. 20, p. 7080-7092, (2020).

Modelling the effectiveness of cathodic protection inthe annular region of submarine flexible pipes

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The production of oil and gas in deep waters requires the use of highly sophisticated equipment, and efficient forms of protection to ensure its integrity. Damage to the outer layer of flexible pipes can lead to marine electrolyte permeation into the metallic (annular) region, as well as water vapor along with gases such as H2S and CO2 can also permeate from the internal fluid toward the annulus. Under any circumstances, flooding of the annular region results in a shorter operational life due to corrosion-related failure mechanisms [1], and the effectiveness of cathodic protection over these structures becomes essential. There is still no consensus on the classification criteria regarding the permeability of cathodic protection along flexible pipes since there is currently no adequate technique that mainly allows monitoring corrosion in restricted regions such as the annular section [2]. In this work, computational models were developed using the COMSOL Multiphysics® software to evaluate the range of cathodic protection in the annular region exposed to the permeation of aggressive species was evaluated. It was possible to assess the current and potential distribution at each point of the flexible pipe. A correlation between the influence of species that permeate the annular region and the effectiveness of cathodic protection was acquired, considering parameters such as solubility, particle , and diffusibility. The results allow the improvement of corrective measures to prolong the lifetime of these components.

Acknowledgments
The authors would like to acknowledge LACTEC and UFPR for their useful cooperation.

References

[1] M. Xua, C. N. C. Lamb, D. Wongb, E. Asselina. Corrosion Science, v. 99, p. 249-257 (2020).
[2] S. Ahmed, V. Orth, M. Foley, K. Kottas, C. Teodoriu. Journal of Natural Gas Science and Engineering, v. 94, p. 4116 (2021).

Morphological stability of Al7Si2.5Cu alloy processed via ultrasonic melt treatment for thixoforming process application

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Developed in the 1970s, semi-solid metal processing has received considerable interest in recent years due to advantages over manufacturing processes like casting. Furthermore, obtaining an alloy with refined microstructure before the thixoforming process is crucial, which is hampered when dealing with aluminum alloys with high silicon content. Therefore, ultrasonic melt treatment (UST) takes place as a possible solution for grain refining. Aiming to evaluate the alloy's morphological stability in the semi-solid state, the melted material was submitted to ultrasonic melt treatment for 20 seconds. After that, the ingots produced were processed via reheating heat treatment at 572°C (45% of solid fraction) for 0, 30, 90,and 210 seconds. Resulting in an average size of primary globules around 88μm, and a circularity form factor above 0.50, it is concluded that UST is an effective alternative for the thixoforming process.

Acknowledgment:
The authors would like to thank the Department of Manufacturing Engineering and Materials at the Faculty of Mechanical Engineering at the State University of Campinas - DEMM/FEM/UNICAMP and the Federal Institute of Education, Science and Technology of São Paulo - IFSP, Bragança Paulista campus.

References:
[1] DE PAULA, L.C., TOKITA, S., KADOI, K., INOUE, H., ZOQUI, E.J., "Analysis of the 355 aluminium alloy microstructure for application in thixoforming", Solid State Phenomena, v. 285, pp. 277-282, 2019.
[2] DE PAULA, L.C., ZOQUI, E.J., "Microstructural refinement of 355 aluminium alloys by ultrasonic melt treatment (UST)", SN Applied Sciences, v. 1, pp. 1-16, 2019.
[3] ATKINSON, H.V., "Modeling the semisolid processing of metallic alloys", Progress in Materials Science, v. 50, pp. 341-412, 2005.

NbC-reinforced Ti composites manufactured by Spark Plasma Sintering (SPS)

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Over the years, there is a growing interest on the use of Ti composites for several technological applications, particularly in the aerospace, automotive, and biomedical device industries. To enhance the mechanical properties of the Ti matrix a variety of reinforcements have been researched, including carbon materials, metal oxides, Kevlar, silicon carbide, and others [1]. In this investigation, the use of NbC as reinforcement was investigated. Morever, the Spark Plasma Sintering (SPS) was tested to consolidate the Ti-NbC composite samples from the mixture of Ti and NbC powders (1:1wt. ratio). Different types of powders were tested to produce these mixture: as-received NbC and Ti powders and cold-rolled Ti and NbC powders. SPS was carried out with heating up to 1000°C, and the samples were kept at this temperature for 3 and 15 min. To assess the mechanical properties and microstructures, microhardness testing and scanning electron microscopy were employed. Samples synthed by SPS showed good consolidation and distribution of NbC particles. Greater microstructural homogeneity was observed for the samples produced with as-received powders and also for the samples in which both NbC and Ti powders were laminated before sintering (SPS with plateau at 1000°C for 3 min). In contrast, in the samples where only one powder was laminated, elongated Ti profiles were observed. This feature was more evident for the sample with Ti:NbC ratio of 1:1. The samples also exhibit significant anisotropy between longitudinal and cross-sections, as observed from microhardness tests. These results indicates that, with adequate selection of the processing parameters, SPS is an interesting route to produce Ti-NbC composites.

Acknowledgments

The authors would like to thank FAPESP (Grant: 2019/22183-6) SFS would like to thank CNPq (Grant: 310984/2020-3) WPS would like to thank CAPES/PrInt Scholarship (Grant: 88887.478283/2020-00).

References

[1] Y. Li et al. Bioactive Materials. 1v(2) 127-131(2016)

NiTi powder sample characterization via reaction sintering for hypersonic airbreathing propulsion system application

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During the operation of a hypersonic vehicle equipped with hypersonic airbreathing propulsion system technology operating over a wide range of flight conditions, it results in high temperatures in the aerodynamic structure causing thermal stresses and rapid ablation. In this context, it was necessary to apply smart materials on the leading edge cowl of the scramjet engine, having a variable geometry that allows the engine to adapt to the different mission profiles faced in its flight [1]. The material chosen for the present work was NiTi (nitinol) alloy, which started with an equiatomic amount of high purity powders,passing through the process of weighing and mechanical mixing in a high-energy planetarymill [2]. Presently, the powder pressing and compacting stage began and finally the reactionsintering process via laser in vacuum. Analysis by secondary ion mass spectrometry revealed that there was variation of oxygen in the layers, regardless of the process parameters, and X-ray diffraction showed the formation of the Ni-Ti system phases and theoxides ($Ni_xTi_yO_w$and Ti_xO_y). Optical microscopy revealed the presence of isolated and interconnected pores of different sizes and morphologies.

Acknowledgements:

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de NívelSuperior - Brasil (CAPES) associated to Project (Process 88887.285953/2018-00 - Pró-defesaIV Grant). Thanks, are also due to the ITA, IEAv, and IME for the support and availability ofthe material and laboratories.

References:

[1] Brito, A. A. R.; Costa F. J.; Passaro A.; Lima M. S. F. “Modeling of shape-memory alloys in scramjet engine applications”, rbav.v40, pp. 1-7. 2020.

[2] Teixeira, R. S. "Produção e Caracterização de Liga Niti Equiatômica por Sinterização aPlasma a partir de Pós Elementares Processados por Moagem de Alta Energia".Tese de doutorado. Instituto Militar de Engenharia, Rio de Janeiro, RJ, 2020.

Open-source code for designing multicomponent alloysfor hydrogen storage

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As an energy carrier, Hydrogen has been studied by the capacity of be a green alternative to energy storage. Among different options of hydrogen storage and transportation, multicomponent alloys has been explored as the large compositional field of these alloys allow the control of the thermodynamic properties of the hydrogen storage. The laboratory of hydrogen in metals at DEMa/UFSCar already has a vast experience in synthesis, characterization, and evaluation of properties of these alloys. To improve the capacity of design alloys for specific applications, a first model to predict thermodynamic properties and the pressure-composition-temperature (PCT) diagram of multicomponent body-centered-alloys (BCC) has proposed by Zepon et. Al. [1]. The elements Mg, Al, Sc, Mn, Fe, Co, Cu, Zn, Mo, and Pd were incorporated into the model and their implementation in open-source code in the Python programming language was made for facilitate the utilization and diffusion of the model [2]. The modeling of systems of alloys reported in the literature were done to demonstrate the utilization of the code as an exploratory tool before the synthesis and characterization of multicomponent BCC alloys.

Acknowledgements: This work is financially supported in part by SerrapilheiraInstitute (grant number Serra-1709-17362), in part by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior-Brasil (CAPES) - Finance Code 001 and in part by CNPq (Project number 132353/2020-2)

References

[1] Zepon,G., Silva,B.H., Zlotea,C., Botta,W.J., Champion,Y. Thermodynamic modelling of hydrogen-multicomponent alloy systems: Calculating pressure-composition-temperature diagrams. Acta Materialia **2021**

[2] Pedroso O. A., Botta,W.J., Zepon,G. An open-source code to calculate pressure-composition-temperature diagrams of multicomponent alloys for hydrogen storage. International Journal of Hydrogen Energy **2022**

Oxidation of protective aluminide coatings at 1000°Cfor ϒ-TiAl alloy by pack cementation

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Titanium alloys are very attractive for utilization in aerospace industries due to their good mechanical properties and low density (4.54 g/cm³). They are recently considered to manufacture turbine components and turbocompressors to supplement Ni-based alloys that, despite their excellent mechanical resistance, have relative high density [1]. However, Ti-based alloys have low oxidation resistance when exposed to high temperatures [2]. Thus, the present work aims to optimize conditions for the formation of titanium aluminide protective coatings on γ-TiAl substrates, with the nominal composition Ti(bal.)-43.5Al-4Nb-1Mo-0.1B (at.%), using the pack cementation technique. Depositions were carried out at 900°C for 4, 9, 16 and 25 hours in vacuum sealed silica vessels.Oxidation resistance of bare and coated substrates was investigated by means of pseudo-isothermal tests carried out at 1000°C for 300 h. Microstructural characterization of thecoatings and the oxidation products was performed by XRD, SEM and EDS. The coatings arecomposed mainly by $TiAl_3$and the mass gains for the coated specimens were six times lowercompared to the bare alloy. A protective layer of $Al_2O_3$was observed for the coated specimens because of the high Al content in the coating ~75 at.%. In contrast, oxidation products formed on the bare γ-TiAl alloy were mainly $TiO_2$and Al_2O_3, non-protective and poorly adherent.

Acknowledgements:

The authors acknowledge the financial support from CNPq (grant nº118967/2022-3).

References:

[1] Inagaki, I.; Shirai Y.; Takechi T.; Ariyasu N.; Nippon Steel & Sumitomo Metal Technical Report, v.106, 22-27, (2014).

[2] Smeltzer, W. W.; Young, D. J.; Progress in Solid State Chemistry, v.10, 17-54, (1975).

Preparation of lanthanum acetate from a mixed rare earths concentrate

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A simple and economical chemical process to obtaining lanthanum acetate of high purity was studied. The raw material in the form of mixed rare earths carbonate 24% $La2O3$ comes from commercial Brazilian monazite. It is used the technique of strong cationic exchange resin, proper to water treatment to the lanthanum''s fractionation and it is achieved a purity of 99.9% $La2O3$ and yield greater than or equal 90%, with the elution of rare earths by EDTA solution at pH 4.0. The complex of EDTA-La is transformed in $La2O3$, subsequently the lanthanum oxide is dissolved in acetic acid to obtain the cerium acetate. The solid salt then is characterized via chemical analysis, thermal analysis, X ray diffraction, infrared spectroscopy and mass spectrometry to certify the lanthanum acetate purity. The analytical data collected allowed to conclude that the stechiometric formula for the compound is $La (CH3COO)3.1.5 H2O$.

References
[1] Queiroz, C.A.S et al, Brazilian Journal of Radiation Sciences, 9 (2021) 01-14.
[2] Queiroz, C. A. S. et al., Journal of Energy and Power Engineering. 9, (2015) 616-621.
[3] Queiroz, C. A. S. et al., Journal of Brazilian Chemical Society. 16, (2005) 1191-1194.
[4] Rocha, S.M.R. ; Queiroz, C.A.S. ; Abrão, A. . Synthesis and characterization of lanthanum acetate for application as a catalyst. Journal of Alloys and Compounds. 344, (2002) 389-393.

Production and Characterization of a CrCoNi Multicomponent Duplex Alloy

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Multicomponent alloys represent a new class of materials that are characterized by not having a single principal element but rather being formed of concentrated solid solutions [1]. These alloys have been the focus of numerous studies due to their unique properties. In this work, we focus on the CrCoNi system, which is an example of an alloy that combines strength, ductility, and corrosion resistance. Our objective was to produce a duplex alloy of this system by choosing a composition predicted by CALPHAD thermodynamic calculations. The resulting alloy was then characterized using various methods, including optical microscopy, electron scanning microscopy, EDS, Vickers microhardness tests, and x-ray diffraction. Our experiments showed that the alloy had both FCC and BCC structures, and that the chemical compositions of these structures were consistent with the values predicted by the thermodynamic calculations. Additionally, we analyzed the effects of cold work on the material. One of the main findings of this work was the absence of the sigma phase in the alloy, which was also consistent with the predictions made by the CALPHAD method.

Acknowledgements: DEMa-UFSCar for the structure and equipment; José Eduardo Spinelli for judging the present work as a graduation monography.

References:[1] F.G. Coury, G. Zepon, C. Bolfarini, Multi-principal Element Alloys From the CrCoNi Family: Outlook and Perspectives, Journal of Materials Research and Technology, volume 15, 2021, Pages 3461-3480, ISSN 2238-7854, https://doi.org/10.1016/j.jmrt.2021.09.095.

Production and Characterization of CrCoNiAl Duplex Multi-Major Element Alloy by Directional Solidification

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Multi-principal element alloys (MPEAs) represent a new class of materials which do not have a major alloying element, being composed of concentrated solid solutions of several different alloying elements [1]. Among these alloys, numerous compositions of the CrCoNi system stand out for having excellent properties, such as strength, ductility and corrosionresistance [2].The development of these alloys has given focus to the search of compositionsthat form face-centered cubic (FCC) structures. However, there are studies that indicate that duplex alloys with microstructures composed of both BCC and FCC phases are capableof presenting excellent mechanical Properties [3]. In this work, an ingot of an MPEA withthe composition Cr44Co35Ni16Al5 will be manufactured. This promising composition for theformation of a duplex structure was chosen based on thermodynamic analyzes and calculations carried out with the Thermo-Calc® software. The ingot of this alloy will bemanufactured through directional solidification using a Bridgman furnace. The objectives ofthis work are: i) the development of the directional solidification process in transient heat extraction regime of the MPEA Cr44Co35Ni16Al5; ii) Microstructural analysis using opticalmicroscopy and scanning electron microscopy (SEM), correlating with cooling rate; and iii)Evaluation of mechanical and corrosion properties resulting from samples solidified underdifferent conditions of cooling rates.

Acknowledgements:

Fundação de Amparo à Pesquisa do Estado de São Paulo - FAPESP (grant 2022/15925-9)and CNPq. This study was financed in part by the Coordenação de Aperfeiçoamento dePessoal de Nível Superior – Brasil (CAPES) – Finance Code 001.

References:

[1] D. B. Miracle, O. N. Senkov, Acta Mater. 2017, 122, 448.

[2] F. G. Coury, K. D. Clarke, C. S. Kiminami, M. J. Kaufman, A. J. Clarke, Sci. Rep. 2018, 8, 8600.

[3] D. Geun, Y. Hee, J. Yang, W. Choi, H. Seop, B. Lee, S. Su, S. Lee, Scr. Mater. 2019, 171, 67.

Production and Characterization of Duplex High Entropy Alloys of the CrCoNiAl System with High Crand Co

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This research presents a study about some high entropy alloys (HEAs) or multi-principal element alloys (MPEAs). Considering the HEAs already produced, the compositions of the CrCoNi system (mainly the equiatomic composition Cr33Co33Ni33) stand out for presenting promising properties with a good combination between resistance and ductility [1].

Most of the studied HEAs are formed of face centered cubic (FCC) structures, but researches have shown that duplex HEAs, also formed by body centered cubic (BCC) phase, have promising mechanical properties [2]. However, little is known about these alloys, because most of the studied alloys containing the BCC phase present the sigma or B2 phase, often presenting brittle and hard behavior, [3], decreasing the interest in studying these compositions.

Thermodynamic calculations were performed to choose four non-equiatomic compositions with high Cr and Co to form duplex structures (FCC/BCC) in CrCoNi and CrCoNiAl systems. Adding aluminum, the duplex field increases on the diagrams, because this element stabilizes the BCC phase. Therefore, it is also possible to add more Co to obtain greater ductility in the alloys.

The alloys were characterized in as cast, homogenized, cold rolled and recrystallized conditions by optical microscopy, X-ray diffraction (XRD) and Vickers micro hardness. Some of the results obtained present dual phase eutectic alloys, another alloy with just one phase, and other results that will help to evaluate the potential and the properties of duplex alloys of the present systems.

Acknowledgements:

We acknowledge financial support from Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq).

References:

[1] F. G. Coury, G. Zepon, C. Bolfarini, Journal of Materials Research and Technology 15, page 1 (2021)

[2] D. Geun, Y. Hee, J. Yang, W. Choi, H. Seop, B. Lee, S. Su, S. Lee, Scr. Mater 171, 67 (2019)

[3] Y. Ma, B. Jiang, C. Li, Q. Wang, C. Dong, P. Liaw, F. Xu, L. Sun, Metals (Basel) 7, 57, page 8 (2017)

Production of Multicomponent Alloys with Good Combinations of Mechanical Properties Selected by a Genetic Algorithm

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Multi-principal element alloys (MPEAs) offer great potential for developing new materials with unique properties that can be tailored to specific applications. However, selecting the right composition can be challenging due to the vastness of the possibilities. To overcome this challenge, the genetic algorithm approach is an effective tool that can generate new alloy families through cross-over and mutation in each new generation, bringing their properties closer to the desired characteristics [1]. A genetic algorithm was created and employed to select two face-centered cubic single-phase multielement alloys based on the parameters VEC and phi while targeting high values of the Hall-Petch constant [2] and the critical resolved shear stress [3]. Their structure was confirmed using thermodynamic calculations via the CALPHAD method, and the alloys were produced using high-purity elements via arc melting. To assess the properties of the selected alloys, Vickers microhardness, optical and scanning electron microscopy, and X-ray diffraction with synchrotron radiation were employed, including in-situ tensile tests. The XRD pattern was also used to calculate the material pair distribution function. These findings were used to validate the genetic algorithm predictions. Overall, the potential of the genetic algorithm is discussed as an approach in the selection of novel materials.

Acknowledgements:

CNPq, DEMa, FAPESP, PPGCEM, UFSCar

References:

[1] Renner, G. Ekárt, A. Genetic algorithms in computer aided design. CAD Comput. Aided Des. 35, 709-726 (2003)

[2] Cordero, Z. C., Knight, B. E. Schuh, C. A. Six decades of the Hall - Petch effect - a surveyof grain- size strengthening studies on pure metals Citation Accessed Citable Link Detailed Terms. Int. Mater. Rev. 61, 495-512 (2016)

[3] Coury, F. G., Clarke, K. D., Kiminami, C. S., Kaufman, M. J. Clarke, A. J. High Throughput Discovery and Design of Strong Multicomponent Metallic Solid Solutions. Sci. Rep. 8, 1-10 (2018)

Recovery of cobalt from used lithium-ion batteries

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Researchers have shown that the Covid-19 pandemic has accelerated several issues, driving the decarbonization of the energy sector. Among these changes, the increased demand for lithium-ion batteries is a highlight, both for use in vehicles and in energy storage for domestic and industrial purposes. Studies show that this continuous increase in demand for batteries can bring environmental problems in the medium term, caused by the accumulation of batteries that arrive at the end of their useful life and are not recycled or recovered [1]. Lithium-ion batteries are rich sources of metals with high commercial value, such as cobalt, lithium, nickel, aluminum and copper. In this context, this research aimed to recover the cobalt contained in lithium-ion batteries, used and discarded, for the synthesis of a cobalt catalyst in the production of biodiesel. The recovery of cobalt from used batteries went through the steps of dismantling, separating the cathode, evaporating the solvents and scraping with a spatula to remove the deposited material, LiCoO2. For the synthesis of the cobalt catalyst, the material underwent acid leaching and subsequent evaporation. Finally, the catalyst was characterized by x-ray diffraction (XRD) and x-ray fluorescence. Biodiesel synthesis was carried out by the transesterification reaction of residual frying oil with methanol [2]. A full factorial design of experiments 22 + P.C. made it possible to obtain the appropriate amounts of methanol and catalyst to be used to maximize the yield of the reaction. Yields greater than 80% were obtained using a methanol/oil molar ratio of 6 and catalyst contents of 1%, demonstrating the effectiveness of the recovered cobalt catalyst for the evaluated reaction.

Acknowledgements:
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References:
[1] N. Gomes, S. Kuber. Global demand for lithium batteries to leap five-fold by 2030- Li-Bridge. Reuters (2023)
[2] R. S. Perez, N.L.C.O. Tapanes, C.A. Feliciano, et al. ACTA SCIENTIAE ET TECHNICAE, v. 4 (2017).

Relaxation and crystal nucleation interplay in supercooled germanium and water

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A well-designed study of the relationship between crystal nucleation and relaxation processes in supercooled liquids could clarify some critical issues in condensed matter science. Using molecular dynamics simulations, we obtained the self-diffusion coefficient, $D(T)$, and the shear viscosity, $\eta(T)$, in supercooled germanium and water, both described viaa Stillinger-Weber potential. Then, three relaxation times were determined 30 % below the equilibrium melting temperature, via i) the shear viscosity, τ_η; ii) the incoherent intermediate scattering function, τ_α; and iii) the self-diffusion coefficient, τ_D. We found that $\tau_\alpha(T) \approx \tau_D(T)$ and $\tau_\eta(T) < \tau_\alpha(T)$, corroborating the findings of recent experimental and computational studies. The birth times of the first critical crystal nucleus at steady-state conditions, $\tau_1(T)$, were obtained both from the seeded and unseeded crystallization data as well as from the Classical Nucleation Theory for different sample sizes. A relevant finding is that the $\tau_\alpha(T)$ and $\tau_1(T)$ curves indeed cross over for germanium and water, confirming the existence of a kinetic spinodal T_{KS} for both liquids. Finally, we demonstrate that if the Kauzmann temperature T_K existed, it would be located well below the T_K; hence, crystallization would intervene in the cooling path before these supercooled liquids could reach T_K, thus averting the Kauzmann entropy paradox.

Acknowledgments: The research is supported by FAPESP grant #2022/05837-5, CNPq and FAPESP contract CEPID #2013/07793-6.

Research progress of transformation products of theZr-rich BCC phase of the Al-V-Zr system

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High entropy alloys containing Al and refractory metals are promising structural materials for aeronautical applications, complementary to Ni-based superalloys. Conceptually, these alloys are preferably composed of at least five metallic elements with concentrations between 5-35 at%. [1]. The knowledge about the phase relations in the Al-V-Zr system corresponds to an important contribution for the development of these alloys, especially for works related to the development of thermodynamic databases. In the present work, the transformation products, during cooling, of the (Zr)-BCC phase, stable in the Al-V-Zr system at temperatures above 800ºC, are being experimentally investigated. Arc-melted samples were heat treated at 1100°C, for 10 days under vacuum, quenched in water, and microstructurally characterized using SEM/EDS and XRD. The results obtained confirm the non-existence of the (Zr)-BCC phase, after quenching, being replaced by the (Zr)-HCP phase, and other products. There are indications of transformations occurring in the solid state (due to the very small size of the precipitates) in the micrographs of all samples. The present results indicate that the accompanying (Zr)-HCP products are either Zr2Al or ZrV2, depending on the chemical composition of the alloy. To investigate in more detail and assert what these products are, microstructural characterizations via EBSD are planned. The results to be obtained may lead to a more precise knowledge of the phase relations in the Zr-rich corner of the Al-V-Zr system, elucidating unanswered questions from previous studies by the research group of which the authors are members.

Acknowledgements:
The authors aknowledge the financial support from São Paulo Research Foundation - FAPESP (grant #2022/06244-8) and CAPES (#88887.663714/2022-00).

References:

[1] Yeh, J.; Chen, S.; Lin, S.; Gan, J.Y.; Chin, T. S.; Shun, T. T.; Tsau, C.H.; Chang, S. Y. Advanced Engineering Materials v. 2, 299-303 (2004).

Scrap aluminum technological recycle for solarcollector applications.

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In the country process of economic development, a driving factor is the development and expansion of the energy sector. Thinking about that, the sustainable production of clean energy has been the focus of several countries. Brazil differs from the world average because its energy matrix is primordially constituted through hydroelectric plants. However, hydroelectric plants are susceptible to the rain seasons. In this context, a diversified energy matrix can minimize the risk of a water crisis affecting the country's development. Wind and solar energy production can be a solution for this problem. The wind electric power generation represents 10,6% and solar electric power is around 2,5% in the Brazil energetic matrix. Increasing the installation of solar collectors in the industrial sector, in the commercial sector and even in residential areas may be a solution to become the Brazilian energetic matrix more diversified. An essential material for the development of solar collectors is the aluminum. Its applicability ranges from structures for fixing solar panels to the box and tubes of heliothermic collectors. Therefore, the research project in question aims to develop aluminum and aluminum alloys through the casting process from recyclable materials for application in solar collectors. The aluminum alloys will be characterized regarding the physical-chemical, corrosion resistance, tribological and mechanical properties of the final goods. The project also aims at the production of technological and scientific knowledge and its dissemination through a partnership that may be signed between teaching and research institutions, CETENS-UFRB and PROCIMM-UESC.

Spectroscopic and Morphological Characterization of MWCNTs/Ti6Al4V/CaF$_2$-Based Flux for Tubular Arc Welding Electrode

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Titanium carbide-based weld coatings are attractive due to their wear resistance. However, the structural and electronic interactions of the flux components of electrodes affect arc stability and the weld surface [1]. Accordingly, this work presents the spectroscopic and morphological characterization of a nanostructured flux that could be used in tubular arc welding electrodes. The flux was produced by mixing multiwalled carbon nanotubes/titanium alloy Ti6Al4V/calcium fluoride (MWCNTs/Ti6Al4V/CaF$_2$), with a mass ratio of 1:40:4, for 1 hour at 30 rpm, using the mechanically allowed method. Scanning electron microscopy results revealed MWCNTs agglomeration around the CaF$_2$crystals and the lamellar microstructures of the Ti6Al4V alpha (α) phase. Based on the X-ray diffraction (XDR) analysis (each component) were identified the following Bragg''s plans: a) MWCNTs (002); b) α-Ti (002) and (101); c) CaF$_2$(111) and (022). In the flux, the intensities of the CaF$_2$peaks highlighted, while those related to MWCNTs and α-Ti reduced. Moreover, the FWHM of MWCNTs (002) and α-Ti (002) increased about by 4% and 39%, respectively, evidencing amorphization by the mixing process, as opposed to the behavior observed for CaF$_2$. The Raman spectrum of the flux showed characteristic bands of CaF$_2$and MWCNTs, where an apparent increase in the degree of amorphous carbon was observed compared to the MWCNTs as received, in accordance with the XRD results. In addition, redshifts in the G and G'' bands suggest microdeformations in the crystal lattices of the inner and outer tubes of the MWCNTs, as well as CaF$_2$-induced n-type doping, which may result in improvements in welding arc stability.

Acknowledgements:
Thanks to CNPq (nº 151500/2021-5), FAPESPA (nº 055/2021) and FINEP (nº 01.22.0133.00) for financial support.

References:
[1] J. G. FAGUNDES JÚNIOR et al. Adição de TiO2, CaCO3 e CaF2 Como Componentes doFluxo de Arames Tubulares para a Formação de TiC. Soldagem Inspeção, v. 25, e2511(2020)

Straining temperature effect on the mechanical and microstructural behavior of austenitic stainless steelAISI 304

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Phase transformations in austenitic stainless steels have been widely studied due to their technological importance. Due to the instability of the austenitic microstructure, it is subject to transforming into martensite during straining processes, which leads to the hardening of these steels. The chemical composition of austenite, the type of straining process, and the straining rate and temperature influence the phase transformation. The TRIP effect (Transformation Induced Plasticity) is much studied, as there is an increase in mechanical strength without significant loss of ductility. The TRIP effect is associated with the (α')-martensite formation, which can pass through a hexagonal-type structure called (ε)-martensite, acting as an (α')-martensite transformation core or nucleating directly from austenite during the straining. In this work, the mechanical behavior of AISI 304 austenitic stainless steels was evaluated by a tensile test at room and cryogenic temperatures. The evolution of (α')-martensite transformation was studied using optical microscopy, hardness measurements, ferritoscopy, and X-ray diffraction in the specimens tested at different strain levels. Based on the results, it is concluded that the straining increases the mechanical strength of the studied steel, and this effect is more pronounced at the cryogenic temperature. In the cryogenic condition, uniform elongation occurs at clear values positioned to the right of the curve, suggesting structural integrity for high strain levels. The increase in strength parameters without significant elongation loss in the cryogenic condition is attributed to the more considerable phase transformation of (α')-martensite from austenite during deformation in the cryogenic condition and may be associated with the TRIP effect.References:[1] I. Tamura. Metal Science, 16(5), 245-253, 1982.[2] Amar K.De et al., Scripta Materialia, 50(12), 1445-1449, 2004.[3] Crivoi, M.R. et al., Cryogenics, 105, 103020, 2020.

Structural and electronic properties of BAs

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In this meeting, we will present the results of our theoretical investigation regarding the structural, mechanical, and electronic properties of cubic boron arsenide (BAs) crystal performed by means of ab initio calculations based on density functional theory (DFT). In our study, BAs was subjected to hydrostatic pressures of 10%, 20%, and 30% to understand how its electronic state changes under the effect of external pressure. The aim of our study was to analyze and describe the change in electronic properties, particularly the occurrence or not of structural and electronic phase transitions at ambient pressure. In this work, all our DFT calculations were implemented using the QUANTUM EXPRESSO (Q.E) computational package. When analyzing the total energy convergence as a function of the lattice parameter, we verify that the lattice parameter is underestimated (overestimated) within the LDA approximation (GGA). The results for the bulk modulus showed good agreement with existing calculations in the literature. The comparative study of the electronic properties of the BAs systems at ambient pressure indicated agreement with the results of previous studies. In this case, the results obtained for the band structure showed that the BAs have an indirect gap between the point $\Gamma - X$ with a value close to 1.15 eV. Analyzing the pressurized BAs showed clear changes in electronic properties, particularly in the high-pressure regime, where its bandwidth is systematically reduced, revealing, in the end, the emergence of a semi-metallic state.

STUDY OF ANDIROBA CAKE POWDER AS CORROSION INHIBITOR FOR CARBON STEEL IN ACID MEDIUM

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Carbon steel is widely used by various industrial sectors and civil society. This is due to characteristics such as low melting point, good weldability, good mechanical resistance and low cost. Despite these advantages, carbon steel has the inconvenience of low corrosion resistance, thus requiring a protection mechanism. Among the strategies aimed at controlling corrosion, the use of corrosion inhibitors is widely chosen. Synthetic chemical inhibitors, such as those from chromium and arsenic, were used due to their good efficiency, however, the harmful effect on the environment and human beings, led to the search for efficient and non-toxic inhibitors, resulting in natural inhibitors of corrosion. Therefore, this work aims to investigate the potential of Amazonian' s fruit residue, Andiroba cake bark, as a corrosion inhibitor on SAE 1008 carbon steel in 1 M HCl. This evaluation was performed using mass loss test and showed that efficiency 70% and 83%, when the concentration of the inhibitor was 1,11g/L and 1,44g/L, respectively. The inhibitor compounds were analyzed by Fourier transform infrared spectroscopy (FTIR) and the presence of hydroxyl groups, aromatic rings and highly electronegative atoms (N and O) often associated with natural inhibitors were observed. This study reveals that the residue obtained from Andiroba has potential to be used as inhibitor corrosion in the replacement to the synthetic inhibitors.

Acknowledgements: FACEPE, FAPESB, LamEQ, UESC.

Study of cutting parameters for top millig of arcsprayed coatings

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The use of coatings to protect mechanical components is a very common practice in industry, because they increase shelf life of components and materials, that originally would not withstand adverse conditions, such as corrosion and wear. Among the different techniques for obtaining these coatings, thermal spraying is widely used due to its relative simplicity and practicality application. The main characteristic of thermal spray coating is its heterogeneous structure, composed of pores, voids, oxides, with a very rough surface. Depending on the application, the sprayed components must pass throught a machining process to smooth the surface and improve the surface finish, which is still a challenge in the industrial sector. Thus, the objective of this work is to analyze the influence of the cutting parameters: cutting speed and feed per tooth, in the end milling of carbon steel pieces coated by thermal arc spray. For this, machining force data, cutting tool wear, surface roughness and surface hardness were analyzed in order to obtain data that characterize the machinability of this material. In addition, a microstructural analysis was carried out using an optical microscope and microhardness measurement to verify how subsurface layers were affected due to the machining process. The results indicate that the combination of higher cutting speed and lower feed per tooth (v_c= 225 m/min and f_z= 0.08 mm), among the researched conditions, presented less roughness and less tool wear. However, tool wear was very severe in all machining conditions studied, reaching or approaching end-of-life criteria normally used, in a machined length of 140 mm.

STUDY OF MATERIALS FOR CRYOGENIC ANTI- SPRINGS THAT WILL COMPOSE NEW GENERATIONS OF LASER INTERFEROMETERS FOR OBSERVING GRAVITATIONAL WAVES

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The Laser Interferometer Gravitational Wave Observatory (LIGO) is an instrument designed to detect gravitational waves. The LIGO detections confirmed the last remaining prediction of General Relativity and ushered in a new era of gravitational wave astrophysics. In order to improve sensitivity by an additional factor of two and halve the low-frequency cutoff to 10Hz, an enhancement to the observatory, dubbed the "LIGO Voyager," is planned. This new interferometer is planned to operate at 123K temperature due to silicon''s zero coefficient of thermal expansion at this temperature. In this sense, it is necessary to adapt the components for operation in cryogenic conditions, as is the case of vibration systems and more specifically their springs. Currently, there are possible candidates to compose these springs, which have mechanical quality factors (Qm) suitable for this type of application, as is the case with the materials: Stainless Steel, Niobium, Tungsten, CuAl (6%), Molybdenum and Aluminum 5052. In order to carry out the measurements under similar operating temperature conditions of the LIGO Voyager, an experimental scheme was developed that allows subjecting the above-mentioned materials to a temperature of 77K. Thus, it was possible to observe the deflection of stainless steel under ambient and cryogenic conditions. As we cool a material, its Young''s modulus tends to increase, for practical purposes, this means that k, which represents the elastic constant in Hooke''s equation, is increased. For stainless steel, for example, it was possible to show that the cryogenic condition increased the spring constant by $1.40*10^2$ N/m.

Study of residual stresses and fracture toughness in welded and repaired joints using A516 Gr70 steel

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The properties of welded joints subjected to cyclic loading is an important subject in several áreas[1]. In this scenario, aiming to study this subject, welded joints made of ASTM A 516GR 70 steel plates, with a thickness of 30.5 mm, welded by the MAG - Metal Active Gas process (20% CO2) with the use of a K-bevel were analyzed. to allow full penetration due to the high thickness [2]. The joints were manufactured with seven welding passes on each side of the chamfer. After welding, one set was subjected to a repair pass, for remelting the surface of the bead using the TIG (Tungsten Inert Gas) process, while another set was subjected to two repair passes. This study aimed to analyze aspects of welded and repaired joints: dimensions (height and width) of the bead, behavior of the residual stress profile measured by X-ray diffraction, Vickers microhardness profile and fatigue crack nucleation with based on ASTM E466 and E606 standards. The results indicated that the greater number of repair passes decreased the compressive residual stress values in the transverse and longitudinal directions, from -350 MPa to 50 MPa. There was greater uniformity in hardness Vickers values (value between 200 and 210 HV) with the use of cord repairs. It was observed that the fracture toughness presented values of 1500 J/mm (without repair) and 900 J/mm (one pass and two repair passes), lower than that found in the material without welding (3500 J/mm). Therefore, as the repair passes were performed, the residual stresses in the weld bead tended to positive values, the hardness tended to stabilize with values of 200 HV due to the increase in the number of repair passes and the fracture toughness decreased in the welded material when compared to the values of the non-welded material. There was no significant difference in fracture toughness between repair passes.

Study of self-healing by electrical pulses with low current flow in damaged aeronautical Al alloy 7075

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High-strength aluminum alloys (7000 series) are commercially applied in strategic aeronautical structural components, but mechanical stresses during use generate microstructural defects that lead to microcracks and failure of a structure. The promotion of self-healing by applying electropulses (EPT - "Electropulsing treatment") is a recent alternative and has the advantage of promoting low heating. Microstructurally, pulsed current promotes stress relief and generates a flow of electrons with a thermal gradient in the region of microcracks that acts to compress their ends and repair them [1, 2]. Samples of a commercial sheet of alloy 7075 were subjected to tensile tests for the production of microstructural defects (grain elongation, microcavities and microcracks), being pre-deformed under different conditions (60%, 70%, 80%, 85%, 90% and 95% of rupture strain). The most severe conditions were submitted to EPT with pulses of 400 amperes, varying the current density up to 50 A/mm2 for 14 s. The microstructural characterization via SEM-FEG was carried out by correlating the damage and the mechanical behavior before and after the electropulse self-healing procedure, being discussed in terms of the effectiveness of the self-healing mechanism in the test conditions used. The applied parameters were effective in promoting partial recovery of microstructural defects with the improvement of the ductility of the samples.

Acknowledgements: Thanks to CAPES, PPGCEM-UFRN and PPG-UFRN.

References:

[1] H. Song, Z. J. Wang, X. D. He, and J. Duan, "Self-healing of damage inside metals triggered by electropulsing stimuli," Sci. Rep., vol. 7, no. 1, pp. 1-11, 2017, doi: 10.1038/s41598-017-06635-9.

[2] Z. Tang, H. Du, L. Lang, S. Jiang, J. Chen, and J. Zhang, "Experimental investigation intothe electropulsing assisted punching process of 2024T4 aluminum alloy sheet," J. Mater. Process. Technol., vol. 253, no. September 2017, pp. 86-98, 2018, doi: 10.1016/j.jmatprotec.2017.11.011.

Study of the biocorrosion of welded joints of aluminum alloys exposed in seawater

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Aluminum is one of the most versatile materials used in engineering, architecture and other industry segments. The expanded use of aluminum and its alloys in industries is due to its high corrosion resistance in some harsh environments such as seawater, however, some welding processes may favor localized corrosion. The objective of this study was to evaluate biofilms formation and biocorrosion process in AA7075 aluminum FSW welded joints in systems containing anaerobic bacteria (SRB), aerobic bacteria (E. coli) and sterile seawater. Static system glass reactors were used for a period of 15 days, being monitored through the quantification of BRS and E. coli (planktonic and sessile), Scanning Electron Microscopy (SEM), Spectroscopy X - Ray (EDX), corrosion rate and gel visualization test. The results showed that there was variation in microbial adherence at the end of 15 days. Corrosion rates were classified as low in the studied systems. The highest corrosion rate value was in the system containing sterile seawater. Specimens submitted to systems containing aerobic and anaerobic bacteria were not affected by the corrosion process within the first 15 days of exposure.

Study of the Hall-Petch relationship for a medium-entropy alloy with carbon addition after severe plastic deformation

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Among the toughest engineering materials known by men are the Cr-Co-Ni face-centered cubic (FCC) alloys. These multi-principal element alloys (MPEAs) have low stacking fault energy (SFE) and can deform plastically by twinning and phase transformation in addition to slip. Studies show that small additions of carbon can increase the strength of CrCoNi with minimal ductility loss. Likewise, grain refinement is also a strengthening mechanism that is notable for good trade-off between both properties. In an effort to combine these phenomena and obtain even higher mechanical properties, we processed a $Cr_{39.6}Co_{30}Ni_{30}C_{0.4}$alloy via high-pressure torsion (HPT). A base alloy without carbon addition, $Cr_{40}Co_{30}Ni_{30}$, was studied concurrently for comparison. Both materials were manufactured through arc-melting of the high purity individual elements. The alloys were thermo-mechanically processed multiple times with cold rolling followed by annealing. Then, the fully recrystallized samples were severely deformed by 5 rotations of HPT with 6 GPa at 1 rpm. A low-temperature heat treatment was employed to induce relaxation of the deformed substructure. Afterwards, the samples were annealed for full recrystallization and minimal grain growth. For all conditions the hardness values were measured with a Vickers microindenter throughout the disk’s diameter. Grain s were measured with electron backscatter diffraction in a scanning electron microscope and in a transmission electron microscopy with the automatic crystal orientation/phase mapping technique. A Hall-Petch (HP) curve was plotted and compared to that of the samples annealed after cold rolling (grain s measured with optical microscopy). The ultra fined-grained alloys resulted in a deviation of the HP relationship

Acknowledgments:

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - 88887.621834/2021-00.

Study of the mechanical properties of the commercial alloys A356 used in the automotive wheels

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Al-Si-Mg system (e.g. A356 alloy) is widely used in manufacturing components for the automotive such as the wheels. When heat treatment (T6) is performed in this alloys, microstructural changes that include homogenization, better distribution of alloying elements and dissolution of soluble phases with Si and Mg and spheroidization of eutectic particles resulting in a notable improvement in mechanical properties.

Based on this premise, this study proposes to explore the microstructure-mechanical properties correlation of the A356 alloy subjected to T6. Different quench rates and artificially aging were used. Initially, the samples were solubilized at 540°C for 5 h. Then, they were immediately immersed in water (quenching) at 30°C, 60°C and 80°C for 2 min. The third step after water quenching, precipitation hardening at 180±3 °C, 200±3 °C and220±3 °C during 5 h is carried out.

The results without T6 showed a phase rich in silicon in lamellae shape, where as in the treated samples, this phase rich in silicon migrated to rodshape. The mechanical tests showed an increase almost 30% in UTS (UTS before T6:167.1±6 MPa; UTS after T6 using quenching of 30 °C: 239.1±7 MPa). The hardness measurements results followed these changes with an increase close to 25% (hardness before T6: 69±3 HV; hardness after T6: 92±4 HV).

SEM analysis showed that coarse silicon particles are modified to smaller spheroidized particles. Upon solubilization, the acicular silicon platelets in the unmodified structure begin to decompose into smaller fragments and gradually spheroidize.

The results indicate that T6 changes the shape of silicon, from lamellar to rods, through a process of fragmentation and spheroidization,providing an increase in hardness and UTS. However, the artificial aging decreases the mechanical properties. This fact suggests that over aging has probably been reached.

Study of the potential application of lemon essential oilas a corrosion inhibitor of the carbon steel in acid médium

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Steels are ferrous alloys widely used in various industrial applications: civil construction, transportation, oil and gas, marine, petrochemicals, automotive. Alloy classified as ASTM A36 is a kind of carbon steel, which has interesting mechanical properties but does not have good corrosion resistance. The high corrosion susceptibility of this steel occurs because, unlike stainless steels, these alloys do not form passive films [1-3]. In view of the costs generated by corrosion and the different applications of carbon steel, several anticorrosive methods have been researched. Recently, a new class of corrosion inhibitors has been studied, the so-called green inhibitors corrosion. These materials have a combination of corrosion inhibiting properties with low toxicity. Recent research has observed that essential oils can be an interesting alternative to classic corrosion inhibitors [1-3]. Considering the extensive use of carbon steel, the costs of corrosion and the need to obtain anticorrosive techniques that have an environmentally friendly behavior, this work evaluated the use of lemon essential oil as a corrosion inhibitor of ASTM A36 steel in acid medium. Immersion tests were used to determine the steel corrosion rate and essential oil inhibition efficiency in 0.1M HCl. The tests were conducted in a naturally aerated medium and at a temperature of 25oC. The results showed that lemon essential oil acted as a steel corrosion inhibitor for the two evaluated concentrations (1000 and 3000 ppm). It was observed that the inhibition efficiency was more significant for the highest evaluated concentration of essential oil.

Acknowledgements:
FAPERJ and UERJ for finantial suport

References:
[1] LOTO R.T., et al, South African Journal of Chemical Engineering, 35, 159-164, 2021.
[2] CHRAKA, A., et al. Materials Today Proceedings, 22, 83-88, 2020.
[3] HOSSAIN, S.M.Z., et al. Arabian Journal for Science and Engineering, 45, 7137-7159, 2020.

Study of the tribological efficiency of DLC thin film coatings on thermoplastic injection mold extractionpins

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Diamond-like carbon (DLC) thin films are amorphous hydrogenated carbon (a-C:H) materials that constitute a surface coating technology, with potential for improving the mechanical and tribological properties of materials [1]. In this study, extraction pins for thermoplastic injection molds, produced in AISI H13 steel, were coated with DLC films using the PECVD (plasma-enhanced chemical vapor deposition) technique, using a silicon interlayer to increase the film adhesion to the substrate [2]. The DLC-coated pins were subjected to a cyclic wear test on a bench device, which simulates the working conditions of the extraction pin when mounted in the injection mold. In the wear test, different lubrication conditions were tested, in order to compare properties and verify the efficiency of the coating.Comparative measurements of sample mass loss after the test, demonstrated that lower wear occurred in samples where the use of lubricating grease was not applied, confirming in practice one of the main properties attributed to DLC films, acting as a solid lubricant due to its low coefficient of friction, thus eliminating the need for lubricating grease [3]. On the other hand, the samples tested with lubricating grease showed a higher wear rate due to the formation of an abrasive paste between the DLC film and the grease during the test. Additionally, scanning electron microscopy (SEM) analysis performed on the surface of the tested pins demonstrated that the film did not exhibit any failures during the test, maintaining adhesion to the substrate, and only wear marks were observable from the test. Moreover, a scratch test analysis was performed on the pin surface, which resulted in high film adhesion values to the substrate and a low coefficient of friction in the range of 0.02. The results obtained demonstrate the efficiency of the coating and prove the possibility of applying DLC to replace the use of lubricating grease in the analyzed components

Surface morphology characterization of oxidized NiTiNb shape memory alloy

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Since the Shape Memory Effect (SME) discovery, scientific interest in this unique property has grown exponentially. NiTi-based are the most commercially important shape memory alloys (SMA) today, due to properties such as high shape recovery and the possibility of becoming biocompatible. Despite the countless researches studying the physical metallurgy of this alloy system, obtaining them inexpensively is still a challenge for industries. One of the costly factors is the need to use an inert atmosphere to prevent oxidation, so it is critically important to study methods to improve the oxidation resistance of NiTi SMAs. One investigation line is the addition of ternary element, where Nb stands out. There are several strategies used to investigate high temperature oxidation behavior, one of them being the characterization of the oxide surface morphology. In this type of analysis, the continuity of the oxide outer layer is verified, as are the shape and dimensions of the grains present. On this basis, the aim of the present work was to verify the influence of 10.5 at.% Nb addition on the surface morphology of NiTi SMAs oxidized at 800 °C for up to 100 h, and at 900 °Cand 1000 °C for 4 h. The results showed that in all cases, except for the binary alloy at 1000 °C (which formed a very porous oxide), the crystals formed are in a faceted shape and the layer is continuous along the alloy surface. With increasing exposure time (from 4 to 100 h at 800 °C) or increasing temperature from 800 °C to 900 °C, it can be seen that the crystalsbecome increasingly thicker, but keep the same geometrical aspect, which suggests maintenance of the oxidation mechanism. At 1000 °C, for both alloys a change ofmechanism seems to have occurred. The ternary alloy exhibits considerably smaller oxidegrains compared to the binary alloy, which indicates high effectiveness of Nb in improvingoxidation resistance.

Surface Plasmon Resonance Spectrometer in the Double Prism Configuration: Fast Characterization of the Dielectric Constant Dispersion of Thin Films

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Surface plasmons are electron collective oscillations in a metal/dielectric interface. Plasmons exhibit very interesting characteristics such as wavelength smaller than the visible photons, propagation length on the micrometer scale and penetration depth into dielectric and metal on the order of nanometers. These remarkable features have been widely applied in reduced-scale devices and biosensing. SPR devices generally are based on the Kretschmann configuration, in which a laser beam is used to excite plasmons in a metal-coated prism surface. However, this configuration is painful (monochromatic) and does not allow for obtaining the dielectric constant and thickness simultaneously. To overcome this problem, we have developed a SPR spectrometer completely automated through the double prism Kretschmann’s configuration. Our SPR spectrometer consists of a halogen lamp witha tungsten filament (300-2000 nm), a motorized rotation stage (minimal step of 0.0003 degrees) containing the double prism configuration and an optical fiber coupled to a portable spectrometer. The double prism configuration simplifies the experimental setup, avoiding adding another rotation stage to the optical system. To calibrate the SPR spectrometer, we initially measured the refractive index dispersion relation (400-1000 nm) of the BK7 prism. After that, putting a nanometric gold thin film (33 nm) on one of the prism's faces, we obtained the SPR curves as a function of the wavelength and incident angle. Through the theoretical model based on Fresnel equations and the iterative Least Squares Method (LSM) it was possible to determine the dispersion (400-1000 nm) of the real (Re{E})} and imaginary (Im{Ɛ}) part of the dielectric constant of the gold film.

Acknowledgments FAPEMIG , CAPES and CNPq.

Synthesis and Characterization of a new Cu(I)-Iodide polynuclear complex with N-Donor ligand for luminescence and thermochromism studies

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In materials chemistry, molecular design is interesting for creating optical devices such as OLEDs or WLEDs and luminescent paints. The coordination chemistry of Cu^I has been extensively studied due to its abundance in results with organic and inorganic ligands, with variations in its structure, ranging from a simple mononuclear complex to extensive coordination networks Complexes based on Cu^I-Iodide nanoclusters (NCs) have drawn attention due to their luminescent properties in the visible region, in addition to their simple synthesis with different ligands, control of their physicochemical properties and excellent optical properties. These NCs have polynuclear Cu^I-Iodide structures coordinated with organic ligands that increase luminescent efficiency, such as rigid aromatic N-donor ligands, showing different conformations and units, the most common of which is the cubane tetramer Cu_4I_4 [1]. The aim of this work is the synthesis and characterization of onecubane tetramer unit complex, already known in the literature [2], $[Cu_4I_4Py_4]$ (Py = pyridine), and a new complex, $[Cu_4I_4TMX_4]$ (TMX = 1,3,7-trimethylxanthine) for analysis of their luminescent and thermochromic properties. The complexes were synthesized and characterized by multiple techniques, displaying a characteristic luminescence when exposed to a 254 nm UV light. As the final goal, the complexes will be used to build up luminescent films in order to study their applicability in optical materials.

Acknowledgments:
UTFPR, LAMAQ, CNPq, CAPES

References:

[1] Troyano, J.; Zamora, F. Chemical Society Reviews, v. 50, p. 4606 (2021)
[2] Fu, Z.; Lin, J. Crystal Growth and Design. v. 16, p. 2322 (2016)

Syringe cell method to study the corrosion resistance of the UNS S32101 lean duplex stainless steel welded by the gas tungsten arc welding double fusion (GTAW-DF)

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This work aims to investigate the corrosion performance of the UNS S32101 lean duplex stainless steel (LDSS) welded by the gas tungsten arc welding double fusion (GTAW-DF). In the study, six welded samples were manufactured with different welding parameters. A Syringe cell was used to characterize the electrochemical behavior of the different welded zones by potentiodynamic polarization tests in NaCl 3,5 % (w.t.) and in a solution of citric acid with addiction of NaCl to simulate the food industry. The results showed that the welding parameters tested significantly affected the corrosion resistance of the LDSS UNS S32101. Besides, a correlation was stablished between microstructure and electrochemical behavior of fusion line (FL), heat affected zone (HAZ) and fusion zone (FZ).

The effect of C and N on the mechanical behavior of medium-Mn steel

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High and medium-Mn steel belongs to the class of Advanced High-Strength Steels (AHSS). The outstanding mechanical properties come from the fully austenitic microstructure which might present induced plasticity mechanisms (TRIP or TWIP) [1]. The stacking fault energy (SFE) is one of the main parameters to describe the deformation mechanism, however, the effect of interstitials is not well assessed in the literature. Only a few studies involving N additions are presented in the literature for this class of alloys and the objective of this study is to observe the effect of the main interstitials (C and N) on the mechanical behavior of medium-Mn steels [2]. Four compositions were designed by thermodynamic models and defined considering the following plan: without interstitials, with C additions, with N additions, and the last with both C and N additions. The samples were produced by arc melting, hot rolling followed by cold rolling and annealing treatment to obtain a homogeneous microstructure. Tensile tests were performed, and it was observed that the alloy in which the austenite was more stable compared to the martensite (with both C and N additions) presented the highest ductility among the studied compositions, and the alloy with only C presented higher ultimate tensile stress.

Acknowledgments:

This study was financed by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001. Brazilian National Council of Scientific andTechnologic Development (CNPq; grant 403955/2021-1 and 166111/2020-1).

References

[1] Yan, S., Liang, T., Wang, Z., Yan, B., Li, T., Liu, X. Novel 1.4 GPa-strength medium-Mn steel with uncompromised high ductility. Materials Science and Engineering A, v. 773, p. 138732, 2020.

[2] Lee, S., Jung, Y., Baik, S., Kim, Y., Kang, M., Woo, W., Lee, Y.-K. The effect of nitrogen on the stacking fault energy in Fe-15Mn-2Cr-0.6C- x N twinning-induced plasticity steels. Scripta Materialia, v. 92, p. 23-26, 2014.

The effect of Co additions in titanium aluminides processed by powder metallurgy

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Titanium aluminides are intermetallic materials with low density and high mechanical properties at high temperatures [1]. Typical processing routes include casting and thermomechanical treatments and are extremely costly. Therefore, less expensive processing techniques such as powder metallurgy become interesting alternatives for these class of materials. One of the greatest challenges of powder metallurgy processing of titanium aluminides corresponds to their high sintering temperatures (>1400 °C) [2]. In this work, processing of Ti-45Al with Co variations between 1-10 at.% was conducted with the aim of lowering the sintering temperatures since Co can induce liquid phase during sintering [3]. The results indicate that there was a systematic decrease in porosity with Co additions and a significant decrease in the sintering temperature. The microstructure changed from fully lamellar with 1 at.% Co to duplex with higher Co additions and an increase in hardness was also observed.

Acknowledgements
This study was supported by the São Paulo Research Foundation (FAPESP), Grant #2018/04564-0 and #2020/04177-6.

References

[1] F. Appel, J.D.H. Paul and M. Oehring, Gamma Titanium Aluminide Alloys: Science and Technology, 1st ed. Wiley-VCH Verlag, Weinheim, Germany, 2011.
[2] J. Soyama, M. Oehring, T. Ebel, K.U. Kainer and F. Pyczak, Sintering Behavior and Microstructure Formation of Titanium Aluminide Alloys Processed by Metal Injection Molding, JOM 69 (2017), pp. 676-682.
[3] Y. Xia, P. Yu, G.B. Schaffer and M. Qian, Cobalt-doped Ti-48Al-2Cr-2Nb alloy fabricated by cold compaction and pressureless sintering, Mater. Sci. Eng. A 574 (2013), pp. 176-185.

Theoretical and experimental investigation on the solidification path of Al-3Cu-xNb alloys (x=0.5, 3 and 5wt.%)

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Nb-added aluminum alloys present in their phase equilibrium diagram thermodynamic uncertainties in the phase transformations during the solidification path [1-3]. In this sense, the main objective of this work was to elaborate an experimental and theoretical study with Al-3Cu-xNb (x=0.5, 3 and 5w.%) alloys in order to obtain the simulated (or theoretical) solidification paths by the respective phase equilibrium diagrams of the investigated alloys, as well as through experimental cooling curves. The thermodynamic Thermo-Calc tool was used to obtain the phase diagrams of these alloys, and a solidification apparatus was developed and used to obtain experimental cooling curves with cooling rates for thermodynamic no-equilibrium conditions, as well as cooling and heating curves using the differential scanning calorimetry (DSC) technique were obtained with cooling rates close to equilibrium conditions. For all studied Nb compositions, the simulations showed the following peritectic reaction (PR): Liq. $\rightarrow Al_3Nb + Al\alpha$, confirmed by experimental equilibrium solidification curves (DSC). On the other hand, microstructure analyzes for non-equilibrium cooling conditions only confirmed the PR for Al-3Cu-xNb alloys (x=3 and 5wt.%).

Acknowledgements:
The authors acknowledge the financial support provided by Federal Institute of Education Science and Technology of Pará, Federal University of Pará, and CNPq - National Council Scientific and Technological Development (Grant 304924/2020-2).

References:

[1] Mendes G, Azevedo H, Dillon T, Rodrigues H, Hoffmann E, Rocha O. Trans Indian Inst Met. 2023. DOI: 10.1007/s12666-022-02863-4
[2] Dillon T, Mendes G, Azevedo H, Rodrigues H, Pereira P, Rocha OTrans Indian Inst Met. 75, pp. 1429-1439, 2022
[3] Schmid-Fetzer R, In Light Metal Systems. Part 2, (ed. Springer Materiais) Effenberg G, Ilyenko S. Springer, Berlin, Heidelberg, pp. 1, 2005.

TiO_2 nanotubes manufactured after Ti35Nb5Ta alloy anodizing for biomedical applications

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The use of titanium alloys as a biomaterial has been commonly applied because they have good mechanical properties, excellent biocompatibility, and excellent corrosion resistance. Despite being a promising material in the biomedical area, health problems due to premature failures, among other complications that alter the functioning of these devices, occur frequently. In this research the technique used aims to work the surface of the alloy increasing the thickness of its natural oxide layer, improving its properties, enabling structural, compositional and morphological changes in the coating of the oxides. The study alloy, Ti35Nb5Ta, submitted to the anodizing process, under controlled thermal behavior, had its samples forged by plastic deformation, through chemical pickling, cutting and weighing, submitted to the arc melting procedure to obtain the alloys. The chemical, structural, microstructural and mechanical characterizations were performed by energy dispersion spectrometry (EDS), X-ray diffraction (XRD), optical microscopy (OM) and scanning electronics (SEM), Vickers microhardness and Young's modulus. It is expected through the analysis of optical microscopy to visualize the of the grain contour as well as its distribution, the presence of elements such as Al,Si,S,Fe in the composition of the alloy by x-ray fluorescence, determination of the β phase via XRD, and how this phase acts on Young's modulus, and the values of microhardness and elasticity, whether they are larger or smaller in comparison to commercially used alloys, and whether there is presence of other structures that may influence the strength of the alloy.

Acknowledgements:
FAPEMA - Fundação de Amparo à Pesquisa e à Ciência - Maranhão

References:
[1] R. Chen, M.D.P. Willcox, K.K.K. Ho, N. Kumar. Biomaterials. 85, 142- 151 (2016)
[2] Chung, H. e Park, T. . Advanced Drug Delivery Reviews, 59, pp. 249-262 (2007)
[3]Bermejo, P. et al. Clinical Oral Implants Research, v. 30, n. 3, p. 229-241 (2019)

Tribological properties of a β titanium alloy processedby angle pressing in equal channels (ECAP)

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Titanium and its alloys are widely used in several industrial sectors, including aerospace, biomaterials, energy, and so on. To address the technological requirements of these applications, new titanium alloys have been continuously developed, mainly β-titanium alloys which are attractive for biomedical and energy-related applications. In this work, the effect of the severe plastic deformation process (SPD) of equal channel angular pressing (ECAP) on the tribological properties of the Ti-29Nb-13Ta-4Mo β-Ti alloy is investigated. In ECAP, a metallic specimen is pressed though a metallic die containing two channels, equal in cross section area, intersecting at a die angle, where it is constrained and bent through a sharp angle which imposes high plastic shear strain in material without any reduction in cross-section area. In this work, ECAP processing was performed using a die with 120º between the channels. ECAP was performed up to 8 passes at 350oC by route A (no specimen rotation between the ECAP passes). The tribological properties of as-received and ECAP processed samples was performed using a reciprocating nanotribometer Anton Paar - NTR³. The interplay between the alloys' microstructures, ECAP processing, and tribological properties are discussed in this work.

Acknowledgements:

Authors acknowledge the financial support from the FAPESP (Grants 19/22183-6) and CNPq (Grant: 310984/2020-3). The Multiuser Central Facilities of the Federal University of ABC (CEM-UFABC) is also acknowledged for facilities access.

Unraveling Short-Range Ordering in CrCoNi Alloys: Insights from Isothermal Treatments and Indirect Detection Techniques.

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Multi-principal element alloys (MPEAs) are a new class of materials that are characterized by the absence of a primary element in their composition [1]. The Cr33Co33Ni33 (at.%) alloy, derived from the Cantor alloy (Cr20Co20Ni20Mn20Fe20 (at.%)), stands out in the literature due to its excellent mechanical properties at room and cryogenic temperatures [1]. However, there are contributions to the mechanical properties of the Cr33Co33Ni33 alloy that have not been fully elucidated. One of the possible contributions is Short-range ordering (SRO). This phenomenon can be defined as the coordination preference of a certain constituent element in the alloy by another, affecting up to 2 atoms relative to the central atom [2]. Due to the small scale of SRO formation, which is in the nanometer range, this phenomenon presents difficulties in detection, generating ambiguities in the literature. Therefore, the present work aims to assess the formation of SRO in 2 compositions Cr33Co(33-x)Ni(33+x) (x=0, 33) through isothermal treatments at low temperatures (475 °C) for different times. The goal is to verify, through indirect detection techniques (thermal capacity analysis, microhardness, and synchrotron DRX), whether the SRO phenomenon occurs, complementing the literature with more information about the SRO for the CrCoNi system.

Acknowledgements:This study was financed in part by the Coordenação de Aperfeiçoamentode Pessoal de Nível Superior - Brasil (CAPES) - 88887.671622/2022-00. Grant 2023/2022/12641-0, São Paulo Research Foundation (FAPESP). Structurally supported by Laboratório de Caracterização Estrutural (LCE).

References:

[1] F. G. Coury, G. Zepon, and C. Bolfarini, “Multi-principal element alloys from the CrCoNi family: outlook and perspectives,” J. Mater. Res. Technol., vol. 15, pp. 3461-3480, Nov. 2021.
[2] L. Zhou et al., “Atomic-scale evidence of chemical short-range order in CrCoNi medium-entropy alloy,” Acta Mater., vol. 224, p. 117490, Feb. 2022.

Unsteady-state Solidification Thermal Parameters Affecting Growth of Primary Dendritic Arms in anAlCuNb Alloy

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Among the various aluminum alloys registered today, we can highlight those that use copper, usually added in levels between 1 and 5%, promoting an increase in hardness and mechanical strength of aluminum alloys, in addition to allowing them to be heat treatable [1]. Niobium is a soft, ductile metallic element, with a specific mass of 8.57g/cm³ and a melting point of 2,468°C. It also has good resistance to corrosion due to the formation of a superficial oxide film responsible for its passivation [2]. Considering the aforementioned importance of Al-Cu alloys, and observing the continuous development of research related to niobium and its importance for Brazil, this work has as main goal to evaluate the effects of thermal solidification parameters, such as growth and cooling (V_L, T_R) on primary dendritic spacings (λ_1) in an Al-3Cu-5Nb (wt.%) alloy, using rates a water-cooled horizontal solidification device. Optical microscopy techniques were used to characterize and quantify the length of the microstructural scale. The $\lambda_1$variation with $V_L$and $T_R$was established by means of mathematical expressions given by the formulas $\lambda_1 = 60(V)_L^{-1.1}$, $\lambda_1 = 214.7(T_R)^{-0.55}$, with the exponent values -1.1 and -0.55 agreeing with those proposed by works carried out in the literature for aluminum-based alloys.

Acknowledgements: The authors acknowledge the financial support provided by Federal Institute of Education Science and Technology of Pará, Federal University of Pará, and CNPq - National Council Scientific and Technological Development (Grant 304924/2020-2).

References:

[1] ABAL - Associação Brasileira de Alumínio. Perfil da Industria Brasileira de Alumínio. 2020. Disponível em: . Acesso em: 15 jul. 2022.

[2] SOUSA, R. ; FERNANDES, L. E. ; GUERRA, W. Nióbio, Revista Química Nova na Escola. v. 35, n 1. P. 68-69, 2013.

Unveiling the Solidification Kinetics of a Hypermonotectic Al-2.3wt%Pb Alloy in a DownwardVertical Device

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Monotectic alloys, especially Al-Pb alloys, have interesting mechanical and thermophysical properties for various industrial applications, such as the manufacture of antifriction materials and electronic components [1]. However, due to the toxicity of lead, more sustainable alternatives are being sought for these applications [2]. The study of aluminum-lead alloys is crucial for identifying their unique properties and developing suitable alternatives. This knowledge can lead to safer, more efficient, and more environmentally friendly products. Thus, to expand studies on Al-Pb alloys and to evaluate the influence of more intense convective flow in the solidification of hypermonotectic alloys, this work aims to study the solidification kinetics of an Al-2.3wt%Pb alloy solidified in a downward vertical device. The gap between the solidified metal and the mold resulted in lower values for both growth and cooling rates compared to similar results in an upward solidification [3], although the directionality of heat extraction was maintained, as observed in the macrostructure.

References:

[1] A.P.Silva; E.S.Freita J.E.Spinelli; A.Garcia; P.R.Goulart. On the growth of the minority phase during downward transient directional solidification of hypomonotectic and monotectic Al-Pb alloys. Journal of Materials Science vol. 47, page 5581-5589 (2012).

[2] N.S.Cosmo; C.A.S.Brabo; S.Q.Mesquita; T.A.P.S.Costa; O.F.L.Rocha; M.A.P.S.Silva. Análise Comparativa do Ângulo de Molhabilidade em uma Liga Livre de Chumbo Sn-2%Sb / Comparative Analysis of the Wetting Angle in a Lead-free Sn-2.0wt%Sb Alloy. Brazilian Journal of Development vol 8, page 13655-13666 (2022).

[3] W.R.Osório; E.S.Freita A.Garcia. Corrosion Performance Based on the Microstructural Array of Al-Based Monotectic Alloys in a NaCl Solution. Journal of Materials Engineering and Performance vol. 23, page 333-341 (2013).

Use of digital image correlation technique in tensile testing of functionally graded materials produced by additive manufacturing.

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Selective laser melting (SLM) is an effective method for producing functionally graded materials. In the SLM technique, metallic powder layers are sequentially deposited onto a substrate and selectively melted using a laser beam to achieve precise control over the geometry and composition of each fabricated layer. It enables the production of multi-material structures with tailorable properties by integrating the structures and functions of various materials [1]. In this new class of materials, the mechanical response, under tensile load, is highly heterogeneous. Therefore, a precise tool is required to obtain quantitative measurements of the locally specific strains. Digital Image Correlation (DIC) has emerged as a viable alternative. DIC is a computational and optical measuring method capable of obtaining deformation and strain fields in multiple scales and has been increasingly used to study stress-strain behavior. This technique requires a set of images recorded during tensile tests and computationally tracing the pixels to aim the displacements through images by the grey intensity of the grid matrix [2]. The aim of this research is to develop a DIC algorithm witha graphical user interface that facilitates its use. To achieve this, open-source technologies and well-known programming languages will be employed. This work also includes the validation of the developed system by comparing it with conventional systems. The developed algorithm was used to perform and analyze tensile tests on various materials. Virtual extensometers were employed to monitor the elastic and plastic deformations of the specimens. The results demonstrate that the digital image correlation technique is a highly effective tool for analyzing functionally graded materials.

Wear-resistant Fe-Cr-Mo-Nb-B coating by plasma transferred arc

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Fe68Cr8Mo4Nb4B16 gas-atomized powders were employed to coat carbon steel substrates through the Plasma Transferred Arc (PTA) process. Four processing conditions were considered: A, B, C e D. For condition A and condition B the powder feed rate was 10 g/min and current were 120 A, varying only the particle size range: 106 - 180 μm and 53 - 106 μm, respectively. For condition C and condition D, the powder feed rate was 6 g/min, using powders with a granulometric range of 53 - 180 μm, being 50% powders with a particle sizeof 53 - 106 μm and 50% powders with particle size 106 - 180 μm, changing only the current: 120 A and 180 A respectively. The coatings were evaluated by X-ray diffraction (XRD), scanning electron microscopy (SEM), optical microscopy (OM), differential scanning calorimetry (DSC), transmission electron microscopy (TEM) and confocal microscopy (CM). The Vickers microhardness was analyzed in the top and cross section of the polished coatings. Tribological tests were performed based on the standard (ASTM G133-05) onpolished surfaces (Al2O3 1-μm), in pin-on-plate reciprocal configuration. Preliminary resultsindicated: i) coatings with refined microstructures, composed of M2B - tetragonal hard boride ii) Fe dilution of the coating on the substrate ~ 23%; iii) Vickers Microhardness ~370 HV0.5; iv) and abrasive wear resistance ~ 1.7 x 10-4 mm3N-1m-1. Such results demonstrate that the Fe68Cr8Mo4Nb4B16 alloy is viable to be used in steel coating processes by PTA. Acknowledgments: The authors are grateful for financial support from FAPESP (grant number 2021-08514-0), Coordination for the Improvement of Higher Education Personnel - Brazil (CAPES) - Financing Code 001, and CNPq. They would like tothank the Structural Characterization Laboratory of the Federal University of São Carlos (LCE/DEMa/UFSCar) and the Graduate Program in Science and Engineering (PPGCEM), São Carlos - Federal University of São Carlos (UFSCar).

Wetting, IMC types and IMC layer growth of a Sn-Bi-In alloy in Cu, Ni and Fe-36Ni pads during reflow soldering

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Currently, the development of new lead-free alloys with low melting point for use in soldering reflow operations of electronic components is increasingly widespread. Recent studies [1-3] demonstrated that Sn-based alloys containing In showed significant reductions of either the contact angle or the interfacial growth of the intermetallic compound (IMC). In the present work the effects of 10 wt.% In addition on the wettability and reaction layer growth/nature of the Sn-40wt.%Bi alloy in copper, nickel and Invar (Fe-36Ni) pads were examined. The solder reflow was carried in inert Argon atmosphere, for samples aged to 180 °C at a rate of 10 °C/min and maintained at that temperature for 15 min, followed by cooling to room temperature at approximately the same rate. The samples were aged at a temperature of 120 °C for 120 h. The layers were characterized by optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), and wetting angle measurements. The ternary alloy had a reduction from 49.2±0.2° to 23.6±1.6° in contact angle compared to the binary Sn40wt.%Bi on Cu substrate. In the Ni and Invarsubstrates, the angles were 46.5±2.9° and 40.4±0.9° respectively. Punctual EDS analysis ofthe ternary alloy on Cu substrate revealed the growth of the Cu6(Sn,In)5 IMC, with substitutive incorporation of In in place of Sn in the crystal lattice (7.53at.%). The Ni3Sn4 IMC formed in the Ni/alloy couple showed more regular morphology, with very low interaction with In (0.98 at.%). The percentage growth of the layer thickness up to 120 hwas 6.7%. Finally, on the Invar substrate, the IMC showed thin and uniform features,containing high In content (65.10 at.%).

[1] X Wu, J Wu, X Wang, J Yang, M Xia, B Liu, J Mater Sci 55, 3092 (2020).
[2] M. Kamal, M. S. Meikhail, A. B. El-Bediwi, El-Said Gouda, Radiation Effects & Defects in Solids, 160, 301-312 (2005).
[3] A. Nabihah, M.S. Nurulakmal., Materials Today: Proceedings 17, 803-809 (2019).

O-Recent Advances in Cold Plasmas and Related Applications

Active diagnostics of electrolytic plasma processes for surface engineering of advanced materials

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Electrolytic plasmas are of increasing interest for many applications, including surface engineering of advanced materials. Important plasma properties that determine processing efficiency depend on charge transfer pathways at the electrode-electrolyte interface that occur under complex polarisation conditions. To understand these pathways, we developed original methods of real-time electrical diagnostics, such as in-situ impedance spectroscopy, pulse transient analysis and dynamic voltammetry, and applied them to a group of electrolytic plasma processes, including Plasma Electrolytic Oxidation (PEO). New data obtained reveals kinetically driven effects associated with both solvent electrolysis and chemical rearrangements in the surface layer, which allows transition from microarc to soft sparking regime during PEO processing of valve metals to be explained. Emerging evidence indicates that dynamic anodic behaviour of these metals during pulse-reverse polarisation is influenced by both prior cathodic biasing and spontaneous processes taking place under unpolarised conditions. This implies the existence of quasi-equilibrium states with characteristic time constants of the order of tens to hundreds of milliseconds that can be attributed to quasi-reversible hydrogen intercalation into the anodic oxide. The insights achieved allow practical recommendations to be made for the rational design of intelligent electrolytic plasma processes providing controllable growth of PEO coatings on complex shape components and enabling PEO processing of metal substrates with large surface area.

A low-cost atmospheric pressure plasma jet for applications in oral medicine

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In the last decade, non-thermal atmospheric pressure plasma jets (APPJs) have been extensively investigated as an alternative therapeutic option for diverse biomedical procedures. Nowadays, plasma medicine encompasses a wide scope of applications ranging from microbial inactivation to cancer treatment and wound healing [1]. The necessity of treating areas of difficult access, such as inside human body cavities, motivated the development of APPJ generated at the extremity of a long flexible plastic tube [2]. In this way, the reactive species produced by the plasma can be safely delivered to a chosen spot, leading to a novel plasma jet application field called plasma endoscopy [3]. This work reports on the study of a low-cost plasma jet device developed at FEG-UNESP that is mostly aimed at applications in dentistry.

Acknowledgements: This work was supported by FAPESP under grant 2019/05856-7.

References:

[1] M. Laroussi, Plasmas, 1, 47 (2018)

[2] E. Robert, E. Barbosa, S. Dozias, M. Vandamme, C. Cachoncinlle, R. Viladrosa, J. M. Pouvesle, Plasma Process. Polym., 6, 795 (2009)

[3] M. Thulliez, O. Bastin, A. Remy, A. Nonclercq, J. Deviére, A. Delchambre, F. Reniers, J. Phys. D: Applied Phys. 55, 415202 (2022)

Dry Coatings by Cold Plasma for the Innovation in Regenerative and Reparative Medicine

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Over the last 50 years, biomaterials, prostheses and implants saved and prolonged the life of millions of humans around the globe. Today, nano-biotechnology, nanomaterials and surface modifications provide a new insight to the current problem of biomaterial complications, and even allows us to envisage strategies for the organ shortage. In this talk, creative strategies for modifying and engineering the surface and the interface of biomaterials, including metals, polymers from natural and synthetic sources, will be discussed. The unique potential of low-pressure low-temperature plasma surface modification will be detailed with the overall aim to envisage today how far innovation can bring tomorrowsolutions for reparative and regenerative medicine. Applications for health will be emphasized, including biologically active-based, biomimetic, low-fouling, bactericidal, and antiviral coatings.

References

1. Linda Bonilla-Gameros, et al. The oxidation state of Ag nanoparticles highly affects the release of Ag ions without compromising the mechanical performance and the safety of amorphous hydrogenated carbon coatings. Diamond and Related Materials, 2022.
2. Gabriel Morand, et al. On the adhesion of diamond-like carbon coatings deposited by low-pressure plasma on 316L stainless steel. Surface and Interface Analysis, 2021.
3. Samira Ravanbakhsh, et al. The impact of structure and temperature on the mechanical properties and radiopacity of Ta-W coatings for tiny endovascular medical implants. Materials Chemistry and Physics, 2023.
4. Letícia Marin de Andrade, et al. Surface processing for iron-based degradable alloys: A preliminary study on the importance of acid pickling. Bioactive Materials, 2022.

From cell permeabilization to cosmetic ingredient penetration in skin explants with non-thermal plasmas

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This work reports on the demonstration of the penetration of cosmetic active ingredients (caffeine and hyaluronic acid) in human skin explants following safe and controlled plasma jet exposure. First, temperature, current and biological markers fluorescence associated with various proteins in the stratum corneum and epidermis were measured to check the safe delivery of plasma jet and to select two operation regimes at 1 and 20 kHz. Plasma exposure for tens of seconds is shown to induce a transient modulation of pH, transepidermal water loss and water contact angle revealing a reversible skin barrier function disturbance. Then it is demonstrated that plasma exposure first significantly accelerates the penetration of active ingredients, detected about 30 mn after their topical application following plasma exposure instead of about 6 hours in the control samples not exposed to plasma. Second the tuning of the plasma jet pulse repetition rate allows to control the penetration kinetics. Such ex vivo results agree with previous in vitro experiments [1] also exhibiting a transient permeabilization time window. To the best of our knowledge, this work is one the very first demonstration, beyond preliminary investigation by [2], of safe and controlled plasma-assisted active ingredients skin penetration in the context of cosmetic applications.

Acknowledgements:
This work and V.V. were supported by LVMH CIFRE ANRT n° 2017/0743. This work was supported by CNRS GDR 2025 HAPPYBIO. The authors thanks Imabiotech in particular Raphaël Legouffe and David Bonnel for their very valuable technical contribution to HA MS-MALDI imaging. The authors also expressed their sincere appreciation to Sylvianne Schnebert for supporting the development of this work and for her critical analysis of the results.

[1] V. Vijayarangan et al, 589, 119874, International Journal of Pharmaceutics, 589, 119874 (2020)

[2] X. Wen, et al Drug Deliv Transl Res. ;11(3):741-747, DOI: 10.1007/s13346-020-00808-2 (2021)

Metallic nanoparticles plasma synthesis from organometallic precursors

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Our group has developed a novel, environment-friendly method for synthesizing metallic nanoparticles (NPs) from solid organometallic precursors decomposed by plasma. In this talk, we will review various applications of this method, with a focus on catalysis in fuel cells. Our approach involves mixing organometallic powders with nanostructured carbon and decomposing them in a low-pressure RF plasma discharge, resulting in a fine dispersion of metallic NPs on the carbon substrate. We use TEM, XRD, and XPS to characterize the morphology, crystallinity, and chemistry of the produced materials, respectively, and assess plasma chemistry with OES.

We will first discuss the synthesis of Pt NPs from three different precursors, which are widely used in proton exchange membrane fuel cells. Next, we will present the synthesis of Ni NPs in inert, reducing, and oxidizing plasma conditions. In the third part, we will describe our work on producing bimetallic Ni-Pt NPs [1] to decrease the amount of Pt needed for catalysis, using a single-step or a two-step plasma process. Small, finely dispersed bimetallic NPs were obtained in the two-step process, combining low and high energy treatments. Finally, we will briefly showcase other NP syntheses, including ZnO for photocatalysis, ferromagnetic Fe_xN, PtCu alloys, and Au NPs on TiO_2.

Overall, organometallic plasma decomposition is a versatile way to produce a variety of metallic NPs, including alloys, thanks to the multiple plasma parameters that can be adjusted, such as chemistry, power, time, pulse rate, and single or multiple steps. This one-pot process is fast, solvent-free, and consumes little energy, making it a promising method for NP synthesis.

Reference:

[1] M. Da Silva Pires et al., Plasma Process Polym. 16, 1800203 (2019)

OPPORTUNITIES OFFERED BY SUSTAINABLE DEVELOPMENT GOALSIN PLASMA SURFACE TREATMENTS OF METALLIC SURFACES

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The Sustainable Development Goals (SDGs), also known as the Global Goals, were adopted by the United Nations (UN) in 2015 to ensure that by 2030 all people enjoy peace and prosperity. Materials-related issues can be found in many of the 17 sustainable development goals. In this context, surface engineering not only makes it possible to confer new properties on materials, but also to reduce volumes and masses of materials for a given application. Most of the surface treatments currently carried out in metal-related industries are realized in the liquid phase. The context of the Registration, Evaluation and Authorization of Chemical Substances (REACH) legislation leads many manufacturers to take an interest in gas phase surface treatment like plasma surface treatment or in less hazardous liquid phase like plasma electrolytic oxidation compared to anodizing.

In this presentation, we will give some examples of surface treatment by plasma applied to different SDGs. Surface treatments are found in a number of SDG. It is in SDG 7: affordable and clean energy that great opportunities arise and will arise for plasma assisted surface treatment of metals. We will discuss surface treatments in power plants for energy production (nuclear, solar), surface treatment for corrosion and wear sensitive materials, surface treatments for the hydrogen economy. Surface treatments for thermal management will also be developed in this presentation.

The case of SDG 12: responsible consumption and production is particularly important for surface treatments that must integrate the life cycle assessment. A relevant issue for surface treated products is the end-of-life or demanufacturing. In most cases, the recycling of surface treated products is difficult and the cost for recycling is high. The surface treatments of the future will have to integrate recycling aspects

Use of Pulsed Plasma in Science and Industry

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Pulsed plasma used to process advanced materials is not a novelty for the science, since it has been studied for some 4 decades. A lot of advantages have been experienced by materials when pulsed plasma processed, such as mechanical and physical properties' improvements. In this scenario, some discussion raises up in this interesting topic upon the pulsed plasma processing of materials focused on Pulsed plasma Sintering - SPS, by considering the evolution of this manufacturing technique, scientific approaches, discussion upon the existence of micro-plasma, case studies, and the use of SPS to manufacture parts in industry, considering the adoption of the 4.0 Industry premises.

.

Keywords: Pulsed plasma; Material sintering; Applications in industry, Micro-plasmas

What plasma sources are suitable for my selected applications?

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In this paper we discuss different methods used for the generation of non-thermal, low-temperature plasmas with special emphasis on plasmas sources developed at LabPlasma/UDESC/Brazil. We also discuss technical achievement of plasma sources for different technological applications. Among the plasmas described include a new Asymmetric Bipolar Plasma Power Supply (ABiPPS), adjustable in frequency, amplitude, and pulse width for the generation of plasmas with high energy electrons. The increase in the electrons average energy is achieved by intense positive pulses of short duration (in order of nanoseconds) that increase the electrodes secondary electrons emission, allowing controlled plasma ionization amplification. The positive pulse intensity adjustment also allows the control of the ionized mass volume, besides plasma generation with low negative polarization values. The ABiPPS system allow us to controll some plasma parameters in a independent way, which make this system suitable for several applications, namely, plasma sputtering, surface treatment, nanoparticles functionalization, plasma polymerization, etching, surface functionalization of polymers and ceramics, besides other. Others plasma sources developed at Labplasma will be presented, namely, Thin Mesh Cathodic Cage Plasma and DBD plasma for nanoparticles functionalization.

Fundings: Projeto PAP FAPESC-UDESC 2021TR874 and PRONEM FAPESC-CNPq-2020TR730

Development of a plasma-based platform for Ag-controlled release profile for antibacterial applications

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Due to the increase of hospital-acquired infections, mainly caused by bacterial adhesion on surfaces, antibacterial (AB) coatings have received increased attention [1]. In this regard, Ag-based AB coatings are of interest since Ag has a broad-spectrum AB action, without risk of bacterial resistance [2]. However, their success as long-term AB coating relies on controlling the release of Ag+ [3]. To reach this goal, plasma processes have emerged as a promising method. Thus, this study focussed on low-pressure (LP) plasma processes to deposit Ag-diamond like carbon (DLC) coatings with tunable release kinetics. Special attention was paid to Ag NPs concentration and oxidation state, known to impact Ag release. To improve and modulate the long-term release, a hydrophobic fluoropolymer was added.

The DLC, Ag-DLC, and AgO-DLC coatings were prepared using a plasma enhanced chemical vapor deposition - physical vapor deposition (PECVD-PVD) reactor. Then, the deposition of the fluoropolymer (CFx) coating was performed using a pulsed plasma polymerization reactor. The composition of the films and the release of Ag+ were assessed. The results showed that by changing the power applied on PVD, Ag content was modulated from 2% to 15%, increasing Ag release up to 20 times. The kinetic release was impacted with Ag oxidation state and top barrier: Ag-DLC showed a slow release reaching a concentration plateau, AgO-DLC a fast continuous-release (better dissolution rate), while CFx barrier displayed a slowly release without reaching a plateau.

In conclusion, by combining these LP plasma approaches, it is possible to tune the release profile of the coating, and so its AB activity. Acknowledgements: The authors thank the Natural Sciences and Engineering Research Council of Canada for financial support.

References

[1] Cloutier, et al., Trends Biotechnol 2015 (33) 637.
[2] Khan, et al., J Trop Biomed 2015 (5) 509.
[3] Bonilla-Gameros, et al., Nanomedicine:NBM 2020 (24) 102142.

Development of a visual UV-sensitive polymeric colorimetric device

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Ultraviolet (UV) light and oxygen act on several materials as degrading agents. In polymers, this action may generate irreversible changes, which demand total or partial replacement of polymeric products. A simple way of prediction of their degradation level could help the users of this products. Thus, polypropylene samples were coated with thin organosilicone film, to investigate the discoloration mechanism and the potential of the coating as an UV colorimetric sensor. The samples were submitted to Plasma Enhanced Chemical Vapor Deposition (PECVD), in an atmosphere with acetylene, oxygen, and hexamethyldisiloxane (40:20:40), and total pressure of 23.0 Pa. Radio frequency of 13.56 MHz was applied to the sample holder with power of 40 W for 30 min, leading to an amber color coating, characteristic of double conjugated bonds. Subsequently, the samples were exposed to UV-C light until 42 hours, monitored every 3 hours. The films showed a gradual color change and became colorless after 30 hours of exposition. Ultraviolet-Visible Spectroscopy (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR), Surface Plasmon Resonance (SPR) and image analyses of photography by Hue-Saturation-Lightness (HSL) were used to characterize the samples along photodegradation time. The morphology before and after this process was analyzed by Scanning Electron Microscopy (SEM), and thickness and roughness were measured by profilometry. While HSL and visual inspection could follow the film color changes until 30 hours, UV-Vis and SPR results showed that the degradation kinetics can be well fitted by pseudo-first order model until 42 hours. At this exposition time, the films turned on rougher with a heterogeneous surface. The supposed degradation mechanism is associated with double and triple bonds braking and silicon consumption, followed by atmospheric oxygen incorporation.

Acknowledgments:
CAPES (Brasil) - Finance Code 001 and FAPESP (process 2016/24936-3, 2016/19387-0, and 18/04061-8).

Effect of plasma deposition parameters on properties of TiO2 coatings for antibacterial applications

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Antibacterial (AB) coatings are essential for preventing, controlling and managing the spread of infectious diseases, particularly in medical devices [1]. Among the different AB strategies, metal oxide coatings appear as a promising approach, since metal nanoparticles are known for their AB actions [2]. Plasma-enhanced chemical vapor deposition (PECVD) is a well-established technique used to deposit highly adherent metal oxide coatings [3]. In this study, the effect of deposition parameters on the overall properties TiO2 coatings was investigated.

The coatings were deposited using a PECVD330 reactor (Plasmionique Inc.), with Ti-isopropoxide utilized as the source of titanium (Ti). Two working gases, Ar and N2, were used, and the depositions were performed at different pressures and powers. The in-situ plasma composition was analyzed by optical emission spectroscopy (OES). The coatings topography, hydrophilicity, and surface composition analyzed by atomic force microscopy (AFM), water contact angle and X-ray photoelectron spectroscopy (XPS), respectively. OES results evidenced the presence of Ti ions inside the plasma discharge. AFM and water contact angle results showed that the coatings were uniform and hydrophilic, independent of parameters and gases. XPS results evidenced the impact of the working gas. Indeed, with Ar gas 16% of Ti were detected while only 12% with N, but all displaying the expected stoichiometric composition of TiO2. In conclusion, TiO2 coatings with tailored composition were effectively deposited by PECVD. Future works will be to assess stability, cohesiveness, AB and biocompatibility properties of the coatings.

Acknowledgement: This work was supported by NSERC-Canada-Alliance and Prima-Quebec- partnership funds. DM holds a Canada Research Chair Tier I (2012-2026).

References

[1] Y. Zhan et al. Adv. Eng. Mater. 2022, 24, 1
[2] M. Molaei et al. Ceram. Int. 2014, 48, 21005
[3] A. Jedrzejczak et al. Thin Solid Films 2020, 693, 137697

Influence of the working gas on the temperature of atmospheric pressure plasma jets

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Helium (He) is one of the gases commonly used as the carrier gas to generate cold atmospheric pressure plasma jets (CAPPJs). In this work, it was found that most of the heating of a He CAPPJ comes just from the expansion of the He gas into ambient air. This was observed by measuring temperature (T) values directly in the gas stream and also with the He flow impinging on a copper plate, comparing the conditions with and without discharge ignition. It was found that the T values increased as the distance from the gas outlet was enlarged, even without discharge ignition. Some works have reported a depression in agar substrates when He is the working gas, and it is flushed at high speeds. In a recent experiment in our labs, argon (Ar) and He were flushed with and without discharge ignition on agar substrates at a flow rate of 2.0 l/min. The results have shown that the depression on the material surface occurred only when using He. Curiously, such depression was observed not only when the plasma jet was ignited but also when there was only the gas flow. This indicates that such depressions may be linked to the T value of He gas impinging on agar substrates. In another experiment in our labs, a He CAPPJ was applied directly onto BEAS-2B cells to evaluate the possible cytotoxic effect of the same plasma jet on epithelial cells derived from the human lung tissue, which is known to be sensitive to intense gas flows. The BEAS-2B cells were exposed to the CAPPJ for 5 min at two working distances (5 and 10 mm) between the plasma outlet and the cells. It was expected to obtain larger cell viability for the larger distance, but the opposite was observed. This result may also be linked to the T increment of the He CAPPJ as distance increases. The correlation between increase in T due to the He flow alone and the biological observations will be discussed.

Acknowledgments:
Work supported by FAPESP (#2019/05856-7, #2020/09481-5, #2021/02680-5 and #2021/14391-8) and CAPES.

In vitro evaluation of the antibacterial activity and cytotoxicity of a helium cold plasma jet on the treatment of tracheal prostheses

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Silicone T-tube prostheses (STP) are the most frequently used alternative for airway rehabilitation of patients with tracheal stenosis, whose prevalence increased significantly due to COVID-19 pandemics. The formation of microbial biofilms inside STP can lead to degradation and clinical complications [1]. Cold plasma jet (CPJ) is a promising technology for biofilm control, due to the reactive oxygen and nitrogen species generated and low cytotoxicity. In this study, the antimicrobial activity of CPJ was analyzed against multispecies biofilm formed on STP specimens. The biofilms were formed by Candida albicans (ATCC18804), Staphylococcus aureus (ATCC 6538), and Pseudomonas aeruginosa (ATCC 15442) (n=9) at 37°C for 48 h at 120 rpm, under aerobiosis. The biofilms were treated for 5 min by helium-CPJ at 0.5 cm of distance, 31.0 kHz frequency, 17.2 kV voltage amplitude, and 2.0 SLM gas flow. After plasma exposure, the samples were sonicated in sterile saline solution. Finally, the suspensions were serially diluted and plated in specific agar to determine viable cell counts (CFU/mL). Cytotoxicity against BEAS-2B cells was also assessed. 40,000 cells were plated using LHC-9 medium at 37 °C and 5% CO_2 (n=12). The cells were exposed to the products of CPJ generated inside the T-tube. The plasma treatment was performed for 5 min, using the same parameters adopted for antimicrobial activity assays. After 24h, the cell viability was evaluated using the MTT reagent. The resulting density was measured at 570 nm. Non-exposed control group was included. CPJ showed antimicrobial efficacy against C. albicans and S. aureus cells in the multi-species biofilm (Mann-Whitney, p= 0.0001). The reduction of P. aeruginosa cell count was not statistically significant. The protocol was non-cytotoxic to BEAS-2B when CPJ is generated inside the T-tube.

Acknowledgements: FAPESP (2019/05856-7,2021/02680-5), CNPq (308127/2018-8, 309762/2021-9), and Fundação Dante Pazzanese de Cardiologia.

Physical and electrochemical properties of MLG based electrodes surfaces modified by microwave-assisted reactive plasma.

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The reactive plasma technique is often employed to activate the surface of materials chemically and physically. In this study, plasma treatment processes were used in order to modify graphene and graphene/carbon nanotube electrodes. The plasma parameters herein investigated were plasma intensity, partial pressure of oxygen, and reaction time. The surface morphology, the generation of C-C bond defects, surface chemical composition, interfacial free energy, and electrochemical properties were analyzed for the Multilayer Graphene (MLG) electrodes. The treatments effectively promoted the MLG electrode’s surface activation, considering the results obtained, which showed a dependence on those plasma treatment variables. The results suggested that plasma treatment variable adjustments can be used to modulate specific surface properties with potential applicability in electrochemical sensors and supercapacitors. High capacitances were obtained for MLG electrodes treated in the intense plasma region for the longest time experimented (180 s). They were in fact 1450% higher than the non-treated MLG electrode. Supercapacitors composed of MLG and Carbon Nanotubes (CNT) electrodes were exposed to CO2 plasma treatment. These hybrid supercapacitors exhibited a specific capacitance of up to 256 mF cm-2 (603 F g-1), 260% greater than that obtained before the treatment (97.2 mF cm-2). Outstand capacitance retention after 1000 cycles, and high energy and power densities (24.03 μWh cm-2 and 2.16 mW cm-2) were observed for supercapacitors assembled using plasma-treated electrodes.

Acknowledgements

The authors are grateful to Capes, UFTM, CNPq (302582-2021/5), Fapemig (Grant APQ-01359-21), the Brazilian Institute of Science and Technology (INCT) in Carbon Nanomaterials and Nacional de Grafite Ltda. for the financial and material support.

Plasma in the modification of natural polymers

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Due to the variety of species that can be formed in the plasma, as well as different techniques available for plasma application, plasma is being widely used for starch modification. The starch industrial sectors search for economically feasible and easy to implement solutions for modification of starch granules aiming the reduction of gelatinization temperature and stability of the starch paste. Plasma activated water (PAW) isa relatively new field in plasma field and can be cheap and easy to be implemented in large scale. PAW generates no residues and as it contains reactive oxygen and nitrogen species (RONS), can induce oxidation and pH change in the liquid. Long-lived species, such as H2O2, NO2 –, NO3 –, and O3 are observed in the PAW within weeks. Although the numberof papers dealing with the different uses of PAW increased significantly recently, little is being made in the direction of observing materials properties when immersed in PAW. This presentation will show examples of applications of PAW in the modification of lignin, starch and other polysaccarides by plasma, including the formation of nanoparticles.

Antimicrobial hybrid films produced by plasma assisted deposition techniques

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The COVID-19 pandemic has mobilized the world scientific community to find mechanisms to mitigate the human vulnerability to the transmission of diseases by infectious microbes. In this scenario, the development of surfaces with antimicrobial properties is widely examined to avoid the fomite mode of transmission of infectious viruses and bacteria [1,2]. Therefore, this work presents the use of deposition techniques PVD-MS [3] and PACVD to produce an antimicrobial hybrid film, composed of SiO2 and Cu, on an austenitic stainless-steel surface. For this purpose, a Direct Current Plasma reactor with a magnetron sputtering device was designed, constructed, and tested. Then, deposition experiments were performed with variation of the target voltage parameters, sample holder voltage, and silicon precursor cycle. The objective was to investigate the effect of different plasma parameters on the physicochemical properties of the coatings, while sustaining the potential antimicrobial attribute. Then, the films were characterized in terms of chemical composition, thickness, topography, microstructure, and crystalline phases, in order to associate the physicochemical characteristics to deposition parameters. As a result, most of the produced coatings are composed of a significant amount of metallic copper and copper oxide (antimicrobial agents), homogeneously distributed as sub-micrometer spherical particles. In addition, they presented good adhesion quality.

Acknowledgements:
This study was financially supported by CAPES Foundation.

References:
[1] LEMIRE, Joseph A. et al., Nature Reviews Microbiology, v. 11, n. 6, p. 371-384, 2013
[2] IMANI, Sara M. et al., Acs Nano, v. 14, n. 10, p. 12341-12369, 2020.
[3] GUDMUNDSSON, J T., Plasma Sources Science and Technology, v. 29, n. 11, p.1130, 2020.

Application of cold atmospheric pressure plasma jet in the degradation of methylene blue dye in aqueous medium

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The development of new technologies for water treatment has been motivated nowadays due to the growing need to obtain clean water all over the world. Cold atmospheric pressure plasma jet1(CAPPJ) is a technology that can be applied very efficiently to degrade organic pollutants present in water from industrial processes. In this type of discharge, the application of high electric fields can excite, ionize and dissociate molecules of a gas, producing a very large number of reactive species containing oxygen and nitrogen in the atmosphere surrounding the plasma. These formed reactive species can interact with a material close to plasma2. The objective of this research was investigated the effects of the application of cold atmospheric pressure plasma jet in the degradation of methylene blue dye in aqueous médium using argon as carrier gas. It was possible to evaluate the effects of several parameters such as volume, concentration, pH of the solutions and different irradiation times (60-12000 s). With the use of a UV-visible spectrophotometer was evaluated the degradation of methylene blue dye. An increase in the percentage of methylene blue dye degradation occurred with longer plasma jet application time and, while the pH of the untreated solution was close to neutrality, after treatment the solution acquired an acidic pH. Through the analysis of total organic carbon (TOC) it can be observed that the amount of organic carbon decreased with the time of plasma treatment. The results obtained showed that the degradation of methylene blue dye was strongly dependent of the irradiation time.

Acknowledgment:
The authors thank FAPESP for financial support. Process 2020/06448-7

References:

[1] S. Reuter, T. von Woedtke, K.D. Weltmann, The kINPen-a review on physics and chemistry of the atmospheric pressure plasma jet and its applications, J. Phys. D: Appl. Phys. 51, 233001 (2018).

[2] M. Laroussi, Plasma Medicine: A Brief Introduction. Plasma 1, 47 (2018).

Characterization of silicon thin films using Tris(dimethyl amino)silane by Plasma Enhanced Atomic Layer Deposition

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Thin silicon films deposited by plasma enhanced atomic layer deposition (PEALD) have been extensively studied since they present a variety of high aggregated value applications in microelectronics devices and as protective films for corrosion protection [1, 3]. Amino silane precursors has been used to produce these films by PEALD, but Tris (dimethyl amino) silane (TDMAS) has been reported as a difficult precursor for production of this type of film, due to the high energy required to breakdown the third dimethyl group. In this work, it was investigated the paths of film formation in PEALD using O2 plasma for oxidation of the thermally deposited layers from TDMAS. It was evaluated the influence of the atmosphere composition, on deposition and oxidation stages, on the film properties. The chemical structure and composition of films was analyzed by Infrared Reflection-Adsorption Spectroscopy, Energy Dispersive Spectroscopy and X-ray photoelectron spectroscopy. Corrosion resistance was determined by electrochemical impedance spectroscopy and polarization tests. The results revealed the presence of SiOx, as well as Si-CH3 and Si-N impurities preserved from the precursor' s structure [2, 3]. The atomic proportion of the elements indicated the formation of SiO2, but also confirmed the presence of organic impurities, like CH3. It was observed a uniform interface along the entire film surface that increased the resistance of carbon steel against corrosion in saline environments.

Acknowledgements:The authors would like to thank LNNano/CNPEM for the use of PEALD.

References:

[1] B.B. Burton, et ali, The Journal of Physical Chemistry C, 8249-8257, 113 (2009)
[2] E. C. Rangel et ali, Thin Solid Films, 1561-1567, 515 (4), (2006)
[3] E Maeda et ali, Journal of Vacuum Science Technology A, 032409, 38 (3), (2020).

Cold Plasma Deposition of SiO_2 Films on Polyamide Fabrics Using a Hybrid Corona-Dielectric Barrier Discharge Reactor

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SiO_x films are versatile materials that find numerous applications in different fields such as optical coatings, corrosion protection, and food and pharmaceutical packaging. One of the most commonly used methods for depositing SiO_2 films involves the use of hexamethyldisiloxane (HMDSO) monomer at atmospheric or low pressure. However, this work presents an innovative approach that uses a hybrid corona-Dielectric Barrier Discharge system operating at atmospheric pressure. The system continuously treats conjugated activation and deposition processes, with a total discharge power of 570 W at 56 kHz and employs silicic acid ($Si(OH)_4$) solution as the precursor to deposit SiO_2 films on polyamide fabrics. The use of silicic acid as a cheaper replacement for HMDSO is an attractive alternative that reduces costs without compromising the quality of the deposited films. To analyze the composition and morphology of the deposited films, Fourier Transform Infra-Red (FTIR) spectroscopy and scanning electron microscopy were used, respectively. FTIR analysis revealed noticeable Si-O stretching peaks at 1024 and 1085 cm^{-1}, which became substantially more intense after the plasma deposition process, indicating a higher concentration of nano-silica on the surface. Scanning electron microscopy showed significant erosion on the surface of air plasma-treated fabric and confirms the efficient coating processes of nanostructured SiO_2 films. This innovative approach could have significant implications for the development of more efficient and cost-effective methods for depositing SiO_2 films in different applications, with potential benefits for textile industries.

Acknowledgments: CNPq, CAPES and FAPESP.

Deposition of SiOxCyHz/TiOx Nanocomposite Films on Aluminum Substrate through low pressure plasma

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Titanium dioxide has attracted great attention in the field of environmental purification since it is photocatalytic under ultraviolet light. The photocatalytic efficiency and energy required for inducing the process still represent drawbacks inhibiting the diffusion of the process. In this study a one-step low-pressure plasma methodology for the immobilization of TiO2 particles on silicon-based thin films was investigated. It evaluated the effect of plasma excitation power on the incorporation of TiO2 particles into the silicon-based structure. Substrates were attached at the upper electrode of a capacitively coupled reactor while 0.8g of TiO2 power was spread in the lowermost one. The plasma atmosphere was composed of 20% HMDSO and 80% Argon at a total working pressure of 4.0 Pa. Plasma was ignited and maintained for 3600 s by application of a radiofrequency signal to the lowermost electrode where the TiO2 precursor was placed. The effect of plasma excitation power on the molecular structure and its chemical composition was determined using infrared spectroscopy. Organosilicon to silica films was deposited from varying deposition power in both deposition processes: PECVD and PECVD associated with sputtering. However, titanium was detected in the structures prepared by the hybrid methodology. The proportion of titanium and particles incorporated in the silicon-based matrix was observed to be dependent on the plasma excitation power. An overall decrease in film thickness was observed with increasing power for PECVD and PECVD associated with sputtering deposition. The presence of Ti in the plasma atmospheres changes the plasma deposition mechanisms, thus affecting the deposition rate, roughness, and wettability of the film. Interpretation is proposed in terms of the plasma activation degree and sputtering yield dependencies on the excitation power.

Acknowledgements: The authors thank the Brazilian agencies FAPESP (2017/21034-1) and CNPq-TWAS (190896/2015-9) for financial support.

Development and characterization of structural and tribological behavior of a silicon-doped DLC film using hexamethyldisiloxane

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DLC films are amorphous carbon films consisting of sp^2 (graphite) and sp^3 (diamond) hybridizations. These films have been the focus of recent research due to their low coefficient of friction, and high hardness. However, due to its problem of poor adhesion on metallic substrates and low thermal stability, some alternatives have been developed to overcome that problem, for instance, the doping of DLC films with different elements [1]. This work aims to develop and characterize a Si-doped DLC with hexamethyldisiloxane as the Si propellant, in comparison with conventional DLC tribological and structural properties. Plasma depositions were carried out using a pulsed-DC power supply. Plasma ablation cleaning was performed with a gas mixture of 80%Ar-20%H_2 for 1h. DLC and Si-DLC films were deposited with a gas mixture of 90%CH_4-10%Ar, and 90%CH_4-8%Ar-2%HMDSO respectively, with a gas flow of 30sccm, for 2h. Raman and FTIR spectroscopies were performed to analyze the structures of the films, while fixed ball wear tests with a load of 8N, rotation of 158rpm, and different sliding distances were performedto evaluate the tribological properties of the films. Inspections of Raman spectra reveals that the ID/IG ratio decreased with the presence of Si on DLC, indicating a higher presenceof sp^3 hybridizations and FTIR has indicated the bonding of carbon with silicon. Regardingthe tribological properties of the film, Si-DLC film has presented a lower wear volume, due to the stabilization of sp^3 hybridization. However, the coefficient of friction of the DLC filmhas presented lower when compared to Si-DLC, due to a different wear mode proportionedby this film. Doping the DLC film with silicon from HMDSO has proved itself successfulsince the tribological properties of the film are better than conventional DLC film.

Acknowledgments:
CAPES (001) for the financial support.

References:

[1] - WU, W. J; HON, M. H., 111, 134-140 (1999).

Development of a Practical Fitting Routine to Determine Optical Parameters of Semiconducting ThinFilms using JDOS and Parabolic Band Model

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In this study, we developed a computational routine for theoretical curve fitting of experimental transmittance data of thin films based on the joint density of states (JDOS) [1] model and using the parabolic band approximation for semiconductors. The routine takes experimental transmittance data as input and outputs the best-fit theoretical curve, along with corresponding thin film parameters such as thickness, refractive index, and band-gap energy. Furthermore, this routine incorporates the ability to describe subgap absorption and correct for the effects of film roughness on the transmittance data. To demonstrate its capabilities, we applied the routine to experimental transmittance data of thin films grown by sputtering, including gallium nitride (GaN), aluminum gallium nitride (AlGaN), titanium dioxide (TiO2), and tungsten oxide (WO3), which are of significant interest in various applications such as optoelectronics, catalysis, and energy storage. The obtained results showed good agreement between the theoretical curve and the experimental data for all the materials tested. The thin film parameters, including the thickness, refractive index, and band-gap energy, were consistent with values reported in the literature. Moreover, the subgap absorption and roughness correction also contributed to better agreement between theory and experiment. In summary, our proposed computational routine provides a practical and reliable method for determining thin film parameters and comparing films made of different materials through theoretical curve fitting of experimental transmittancedata of thin films.

[1] S. K. O' Leary, "An analytical density of states and joint density of states analysis of amorphous semiconductors," J. Appl. Phys., vol. 96, no. 7, pp. 3680-3686, 2004.

Acknowledgment
The authors thank the FAPESP (proc. 2015/06241-5) and CNPq (428591/2018-3) agencies for the funding.

Dielectric Barrier Discharge-Assisted Atmospheric Pressure Polymerization of Permethrin on Polyamide Fabric for Effective Insect Control

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This work presents the polymerization of permethrin (15%) based monomers on polyamide fabrics using dielectric barrier discharge (DBD) to enhance insect-parasite repellency functionalities in polyamide fabrics. Before the polymerization process, the polyamide fabric was activated by DBD discharge using a plasma dosage of 2841 W. min. m^{-2}, operating at 23 kHz with a voltage amplitude of 12.5 kV in atmospheric pressure. After that, the polymerization process was initiated by injecting permethrin into the system while maintaining the operational parameters used in the activation process. The non-activated and activated polyamide fabrics were measured for static and dynamic contact angle, which showed a variation from 120° (non-activated) to 34° (immediately after plasma activation).The chemical structure of the synthed permethrin was evaluated by Fourier Transformed Infrared (FT-IR) spectroscopy to confirm the polymerization (deposition) of permethrin on the fabric surface. It was possible to observe that the 648 cm^{-1} bands were associated with the asymmetric vibration of the C-Cl bonds, but the most significant change occurred at 1045 cm^{-1}, which was associated with the cyclopropyl group vibrations. Field Emission Scanning Electron Microscopy (FESEM) analysis was used to evaluate the possible degradation of the fabric surface when exposed to plasma activation and the homogeneity of the permethrin coating in the fibers after the polymerization. The Energy Dispersive Spectrometer (EDS) was used to confirm the polymerization and the distribution of permethrin in the fabric. The study found that a tested repellent showed 100% efficacy against Aedes aegypti, even after undergoing ten washing cycles.

Acknowledgments: CNPq, CAPES and FAPESP

Effect of carbon nanotubes obtained by PECVD in biological systems.

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The carbon nanotube is one of the known carbon nanostructures with attractive electrical, thermal, and mechanical properties for various applications. For this reason, the use of these carbon nanostructures in products has been growing in several areas of activity. On the other hand, these products with nanostructures will be at some point discarded in the environment and may have the interaction of nanostructures with biological systems and generate toxicological effects. Thus, it is important to study the ecotoxicity of these carbon nanostructures. This work aims to study the ecotoxicity of multi-walled carbon nanotubes (MWCNTs) obtained by the Plasma Enhanced Chemical Vapor Deposition (PECVD) technique on the algae *Raphidocelis subcapitata*, bacteria *E. Coli*and *S. epidermidis*, found in the environment. The MWCNTs were grown on the nickel substrate with the precursorgases of methane (CH_4), H_2, and Ar at a total gas pressure of 1.50 Torr, with 700 V for 15 minutes. The carbon nanotubes were characterized by Raman spectrometry, high-resolutionscanning electron microscopy (SEM), and transmission electron microscopy (TEM). The aquatic toxicity test used algae of the species *Raphidocelis subcapitata*, in concentrations of 0.1 to 100 mg/L of MWCNTs. The effect of antibacterial activation was used on strains of *E. Coli*and *S. epidermidis*. The material obtained showed a Raman spectrum with D, G, and 2D bands, characteristic of MWCNTs. The images obtained by SEM and TEM show the nanotubes and the multiple walls are observed. The algae growth inhibition test with commercial MWCNTs showed an $EC_{50}$of 0.7191 mg/L and the MWCNTs obtained by plasma showed an $EC_{50}$of 9.316 mg/L. Microbial tests show that MWCNTs obtained from plasma have no antibiotic effect on the studied bacteria. The results show that the MWCNTs obtained from plasma have low toxicity when interacting with biological systems.

Effect of cold atmospheric argon plasma jet treatmenton titanium surface wettability and protein adsorption

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A wettability of surfaces is a key property that affects their biocompatibility and interaction with biological systems [1, 2]. Plasma treatment can modify the wettability of surfaces by adding polar functional groups, but these changes may not be stable over time [3]. We exposed titanium surfaces to an argon plasma jet produced and measured by standard methods. We evaluated the wettability of titanium by measuring the contact angle and calculating the surface free energy and the interfacial energy for albumin and fibrinogen proteins. We also investigated the effect of aging time on the wettability of the treated surfaces. The plasma jet reduced the contact angle of titanium and this effect persisted for 7 days. The surface free energy and the polar component increased after plasma treatment, indicating an increase in hydrophilicity. The interfacial energy for albumin and fibrinogen decreased after plasma treatment, suggesting an improvement in protein adsorption. Our results demonstrate that argon plasma jet can effectively modify the wettability of titanium and enhance its biocompatibility. This technique has potential applications in biomaterials and implantology.

Acknowledgements:
National Institute of Surface Engineering, CNPq.

References:

[1] Tang, L., Thevenot, P., Hu, W. (2008). Surface chemistry influences implant biocompatibility. Current topics in medicinal chemistry, 8(4), 270-280.

[2] Polini, A., Yang, F. (2017). Physicochemical characterization of nanofiber composites. In Nanofiber composites for biomedical applications (pp. 97-115). Woodhead Publishing.)

[3] Petters, S. S., Pagonis, D., Claflin, M. S., Levin, E. J., Petters, M. D., Ziemann, P. J., Kreidenweis, S. M. (2017). Hygroscopicity of organic compounds as a function of carbon chain length and carboxyl, hydroperoxy, and carbonyl functional groups. The Journal of Physical Chemistry A, 121(27), 5164-5174.

Effect of home-made non-thermal plasma jet on oral mucosal wound healing in rats undergoing chemotherapy treatment: a screening setting

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Head and neck cancer is public health problem worldwide with a prevalence of 1.1 million cases annually [1]. Chemotherapy drugs can cause oral mucositis as a side effect of the drug toxicity. Oncological patients with oral mucositis usually present pain and injury which can enhance the risks of infection [2]. Non-thermal plasmas have stood out for their promising effects in the treatment of wounds [3]. This study aimed to evaluate the action of home-made prototype of cold atmospheric plasma jet (CAPPJ) in the healing of oral mucosa wounds in rats underwent to chemotherapy. This screening study was performed with male rats (*Rathus norvegicus*), which received intramuscularly doses of the chemotherapy drug 5-Fluoruracil (5-FU, 30 mg/kg), followed by oral mucositis induction in the vestibular fornix, with the aid of sterile filter paper soaked in 50% acetic acid. For the treatment, the animals were anesthetized and the wound treated for 2 consecutive days with CAPPJ with helium gas (He) in a time of 5 min at a distance of 1.5 cm. Control group was not exposed to the plasma jet. Images of the vestibular fornix were obtained after 3, 7 and 14 days of the oral mucositis induction (5 per group), followed by the measurement of the injured area in the Software Image J and statistical analysis of the data. Reduction of the injured area was observed after 14 days, with 50.00% reduction for the control group and 78.95% for the CAPJ-treated group. A statistically significant difference was observed between the control (untreated) group after 3 days and the plasma-treated group after 14 days ($p=0.03$; One-way ANOVA, followed by Turkey''s multiple comparison test), with the average of the injured area of 20 mm2 for the control group and 4 mm2 for the treated group. These findings demonstrate the promising capacity of CAPPJ with He gas for treatment of the oral mucositis.

Acknowledgments:This work was supported by FAPESP 2019/25652-7.

References:1-Mody,2021;2-Pereira,2019;3-Garner,2021

Effect of low pressure plasma on N95 respirators contaminated with bacteria

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Face filter respirators are used primarily to prevent the spread of airborne pathogens. The fact that face respirators protect the mouth and nose, prevents the contact of contaminated hands with the nasal and oral mucous membranes. The sterilization of this material must eliminate pathogens transmitted by contact with the skin and hands. In a previous work, it was demonstrated that low pressure plasmas, established from citric acid solution vapor diluted with O2 and N2 (13.56 MHz 100 W), promoted effective disinfection of mask fabrics with fungi. In this work it was proposed to evaluate the effect of low pressure plasma on the bacterium Staphylococcus aureus (S. aureus) ATCC 25923. This bacterium is the main contaminant of the skin and is involved in medium and high severity infections. Plasma was generated by applying a DC voltage in a vapor environment of citric acid diluted in isopropyl alcohol solution. Samples treated on positively and negatively polarized electrodes were investigated. Preliminary results indicated that only citric acid vapor was not able to eliminate S. aureus from the samples. Only for one polypropylene sample, a small decrease in the amount of bacteria present after exposure to citric acid vapor was observed. The results indicate that plasma can be a real possibility for disinfecting face respirators for pathogens transmitted by interpersonal contact.

Effect of multicomponent oxide coatings obtained by PEO on the electrochemical properties of commercially pure titanium for biomedical use

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The inflammatory responses of human body reduce local pH as reaction against supposed infection. Ti implants are submitted to this acidification which can develop pitting over them. This type of degradation is undesirable because it allows metal ions to diffuse through the body and reach levels of toxicity [1]. One way to reduce the possibility of corrosion of an implant is to increase the resistance of its protective oxide film against chemical attacks [2]. In previous studies, zirconium and tantalum have demonstrated positive contributions to increased corrosion resistance in titanium alloys [3]. The conditions experienced by an in vivo implant are complex to reproduce, but a preliminary in vitro evaluation of the corrosion suffered by a biomaterial seems reasonably interesting. This work studies the electrochemical behavior of multicomponent oxide film obtained by plasma electrolytic oxidation with deposition of Zr and Ta oxides on a commercially pure titanium matrix, in a simulated environment of inflammation.

Acknowledgements:
Elidiane Cipriano Rangel

References:
[1] BAHRAMINASAB, M. et al. Corrosíon of Al2O3-Ti composites under inflammatory conditíon in simulated physiological solutíon. Materials Science and Engineering: C, v. 102,p. 200-211, set. 2019.
[2] GENTIL, V. Corrosão. 7. ed. Rio de Janeiro: LTC, 2022.
[3] ZHENG, Y. F. et al. Enhanced corrosíon resistance of Zr coating on biomedical TiNi alloy prepared by plasma immersíon íon implantatíon and depositíon. Applied Surface Science, v. 255, n. 2, p. 512-514, nov. 2008.

Effect of the Plasma Boriding Treatment using Solid Paste on the Surface Properties of AISI 1020, SAE 304 and Ti6Al4V Alloys

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Boriding is a thermochemical treatment that involves boron diffusion on the metal surface and is an effective method to improve the surface properties of metals [1,2]. This work aims to study the effect of the plasma boriding treatment using solid paste at a lower temperature and treatment time compared to the traditional method on the boride layer formation and wear resistance in different types of metal. Plasma treatment was performed using a DC-pulsed source at 750°C for 6 hours in an atmosphere of 40% N_2, 40% H_2, and 20% Ar at 500 sccm of total gas flow. The solid paste composed of boriding agent Ekabor2®, $Na_2B_4O_7$, and ethyl alcohol was sprayed on samples of Ti6Al4V, AISI 1020 steel, and SAE 304 stainless steels. The samples with and without treatment were characterized by microhardness, metallography, and microwear test by fixed ball. The formation of the boride layer was observed in all treated samples without significative variation in hardness compared to base metal due to the thinner formed layers. The formed layer, even thin, was efficient in increasing the wear resistance of all treated materials, being more effective for stainless steel 304. It was observed that the type of substrate influenced the thickness and wear resistance of the treated material, whose boron was more effectively diffused in the titanium alloy, while the FCC structure present in the stainless steel favored the diffusion for the ferrous alloy compared to with the characteristic BCC structure of 1020 steel. It is concluded that the plasma boriding treatment using solid paste was efficient in the boride layer production that increased the wear resistance of the studied metals, whose type of substrate significantly influenced the characteristics of the produced layer.

Acknowledgements:
We would to thanks CEETEPS, CNPQ (163629/2021-8), Capes (code 001), and Fapesp (2021/05995-7).

References:
[1] M. Kul et al. Mater Lett 279, 1-4 (2020).
[2] I. Gunes et al. Mater Des 32, 2380-2386 (2011).

Effect of the Plasma Power on Grounded Electrode on Disinfection of N95 Respirators.

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The work of health professionals entails physical, chemical and biological risks that can compromise their integrity and well-being. To minimize biological risks, face masks play a key role. The sterilization of these respirators has important implications for disposal, requiring reuse and recycling. In a previous work it was demonstrated that low pressure plasmas, established from citric acid solution vapor diluted with O2 and N2 (13.56 MHz 100 W), promoted effective disinfection of mask tissues, demonstrating the potentiality of this new sterilization approach. In this work, the same plasma composition was used in order to evaluated the effect of the plasma excitation power, on the disinfection of N95 mask fabrics (polystyrene and polypropylene) contaminated with fungi Saccharomyces cerevisiae ATCC 9763. Contaminated fabric samples were positioned on the grounded electrode (topmost) of a capacitively coupled reactor. All the disinfection experiments were conducted for 10 min in atmospheres containing 50% of citric acid solution's vapor, 25% of O2 and 25% of N2 at a total pressure of 9.3 Pa. Plasma was ignited by application of radiofrequency signal to the topmost electrode. It was investigated the effect of the plasma excitation power, which was varied from 100 to 300 W, on the properties of the fabric (fibers integrity and wettability), on the fungal growth and on the number of Clonies Forming Unit (CFU). An effective sterilization was observed for polypropylene, while for polystyrene it suffered a fungistatic action. This result was observed in the powers of 150 and 300 W. Despite disinfection efficiency, the plasma excited with 100 W showed the best effective result. Tests with lymphocyte cultures demonstrate that plasma in masks does not generate toxic residues. The evaluation of the results indicates that the plasma treatment can be a new option in the sterilization and reuse of the facial respirators. Since the power to be used should be better investigated.

Effect of the Plasma Power on Lower Electrode on Disinfection of N95 Respirators

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Sterilization of face filtering respirators has important implications as one considers their disposal, the necessity of reuse by scarcity and the recycling of pieces used in high contagious environments. In a previous work it was demonstrated that low pressure plasmas, established from citric acid solution vapor diluted with O2 and N2 (13.56 MHz 100 W), promoted effective disinfection of mask tissues, demonstrating the potentiality of this new sterilization approach. In this work, the same plasma composition was used in order to evaluated the effect of the plasma excitation power, on the disinfection of N95 mask fabrics (polystyrene and polypropylene) contaminated with fungi Saccharomyces cerevisiae ATCC 9763. Contaminated fabric samples were positioned on the lower electrode of a capacitively coupled reactor. All the disinfection experiments were conducted for 10 min in atmospheres containing 50% of citric acid solution's vapor, 25% of O2 and 25% of N2 at a total pressure of 9.3 Pa. Plasma was ignited by application of radiofrequency signal to the lowermost electrode. It was investigated the effect of the plasma excitation power, which was varied from 100 to 300 W, on the fungal growth and on the number of Colonies Forming Unit (CFU). An effective sterilization was observed for the polypropylene, while for polystyrene it suffered a fungistatic action only in power 150 W. The worst effect on the disinfection of the masks was obtained with a power of 300 W. Aside presenting a good disinfection efficiency, the plasma excited with 100 W presents the best coast-efficiency relation and did not affect the fibers integrity. Preliminary tests on lymphocyte cultures indicate that exposure to plasma does not generate toxic residues. A discussion is needed regarding the effect of low pressure plasma power on fungal cultures. Acknowledgment FAPESP grant #2022/08020-0.

Enhancing Coating Adhesion on Metallic Surfacesthrough Cold Plasma Treatment

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Corrosion prevention is often achieved by applying coatings, including polymeric coatings in various forms, such as liquid coatings, powder coatings, or solvent-based coatings, as well as paints, which are typically liquid coatings that contain pigments and binders. However, a critical characteristic that directly influences the coating's performance is its adhesion to the substrate. Our preliminary study aims to enhance coating adhesion by subjecting metallic surfaces - low-carbon steel and aluminum - to cold plasma treatment, which uses ionized gas to remove organic and inorganic contaminants from the surface, leading to a more active surface because of the increase in surface energy. In order to achieve our objective, a cold plasma reactor was employed with the following operating parameters: V = 700 V; P = 1 Torr; t = 10 min of plasma treatment; and a gas mixture of hydrogen at a flow rate of 40 SCCM and argon at 160 SCCM. Rectangular steel and aluminum plates, measuring 90 x 60 mm, were treated at the cathode using the previously mentioned reactor. Following plasma treatment, both treated and untreated samples were coated with a layer of polyurethane varnish applied using a 100 µm spiral bar coater. In accordance with the ABNT NBR 11003, an adhesion test was then performed. After the test a salt solution of 50 g/L was sprayed over the grid pattern described in the standard to evaluate the corrosion resistance. The results showed that the adhesion behavior of the aluminum samples was significantly improved after treatment, whereas the adhesion of the steel samples did not exhibit the same level of improvement. Furthermore, when it comes to corrosion, aluminum samples do not present significant differences between the treated and untreated samples. However, at steel samples, when more adhesion occurred, less corrosion spread under the varnish was observed.

Evaluation of the antitumor effect of NaCl and Ringer lactate solutions treated with atmospheric pressure plasmas on the in vitro viability of melanoma cells.

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Recent literature presents encouraging studies on direct and indirect application of atmospheric pressure plasmas (APP) for the treatment of superficial tumors. The plasmas have been directly applied on tumors or to saline solutions, which are subsequently applied to the tumor mass. Melanoma is one of the most common types of cancer in the world. Particularly, in Brazil it corresponds to about 30% of cases registered in the country annually. In this scenario, the objective of this work was to evaluate the effect of sodium chloride 0.9% NaCl and Ringer lactate solutions exposed to APP on B16F10 murine melanoma cell line in two- and three-dimensional cultures. The solutions were exposed to argon plasmas for 30, 45 and 60 min and sterilized in 0.22 μm filter. After the treatments, the B16F10 cells and the control cell line L929 (murine fibroblast) were incubated for 30, 60 and 120 min with the pristine and plasma treated solutions. Cell viability by MTT has shown that the treated solutions significantly reduced the viability of B16F10 cells when compared to untreated solutions. After the experiments, the solutions were frozen and tested again. It has been observed that even 75 days after the plasma exposure, the solutions were still able to continue reducing B16F10 cell viability. The evaluation of the type of cell death showed that the different solutions induced both cell death by apoptosis and necrosis, positive for Annexin V and/or propidium iodide. As assessed by flow cytometry, the treatments also interfered with the expression of surface molecules, such as PD-1 and PDL-1, responsible for the suppression of the antitumor immune response. Regarding the 3D culture, the capacity of the liquids to reduce the of the spheroids in vitro was evaluated, comparing with the liquids treated on the same day. In conclusion, liquids treated with APP and frozen have presented an effect on tumor cells even after 180 days of treatment. Acknowledgement Fapesp process 2020/06448-7.

Exometabolomics of fibroblast cells on titanium surface modified by cold atmospheric plasma.

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The use of cold atmospheric plasma (CAP) can be use to improve the titanium surface functionality for induces cells to synthe adhesion proteins and activate metabolic pathways [1][2]. Both are crucial for ensuring implant quality and healing; However, it is unclear which metabolic pathways CAP activate. Therefore, our objective is to evaluate the exometabolomics of fibroblasts on CAP-modified titanium surfaces. Titanium discs were polished and treated with CAP at 15kV,600Hz, and a He flow rate of 1 L/min for 15 minutes. The discs were characterize for wettability(sessile drop), roughness profile (AFM), and chemical composition (XPS). 3T3 cells, murine fibroblasts, were cultured in DMEM with 10% fetal bovine serum for cell viability tests (MTT). Additionally, exometabolomic analysis used the protein S precipitation protocol for NMR. We observed that CAP increased the OH-bonds, making the titanium surfaces more hydrophilic (14°±2.2 vs. 59°±1.8, $p<0.01$) andreduced surface roughness (3.6±1.01 vs. 7.5±2.05, $p<0.01$). On the treated surfaces,cellsincreased their viability (0.61±0.1) after 24 hours. The changes in glutamine metabolismconfirmed by cell proliferation, as CAP prefers the aerobic pathway for more efficient energy production and cell adhesion to titanium [3].

Acknowledgements:This study was financed by CNPq (Brasil) - (Protocol 402536/2021-5)And CNPEM(20220649).

References:

[1] T. Bernhardt,M. L. Semmler,M. Schäfer,S. Bekeschus,S. Emmert and L. Boeckmann, Plasma Medicine: Applications of CAP in Dermatology. Oxidative Medicina and Cellular Longevity. 10, 10-13(2019).

[2] C. Jin,Y. Liu,L. Sun,T. Chen,Y. Zhang,A. Zhao,X. Wang,M. Cristau,K. Wang and W. Jia, Metabolic profiling reveal disorder of carbohydrate metabolism in mouse fibroblastic cells inducel by TiO2 nanopaticles. J. Appl.Toxicol. 33, 1442-1450(2013).

[3] E. Johar D,A. Elmehrath,R. Khalil,M. Elberry,S. Zaky,S. Shalabi,L. Bernstein, Protein networks linking warburg and reserve warburg effects to cancer cell metabolism.Biofactors 47, 713-728 (2021).

Friction and wear between steel test bodies using RF plasma-deposited diamond-like carbon flakes dispersedin oil.

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Diamond-Like Carbon (DLC) coatings have been extensively used in the industry to reduce friction and wear.[1] Hydrogenated amorphous carbon (a-C:H) films deposited by plasma discharges on hydrocarbons such as methane and acetylene are the most commonly used films. Recently, it was reported that the coefficient of friction between DLC-coated surfaces can be altered by the presence of DLC particles resulting from the wear of the coating in the lubricant oil.[2] The aim of this study is to investigate the coefficient of friction between steel surfaces coated or not with DLC, using lubricating fluids with an additive based on DLC flakes. The DLC flakes were prepared by depositing DLC films, by RF glow-discharge plasmas (50 mTorr pressure, -300 V self-bias) onto commercial vacuum pump oil, which were added to mineral oil, to form the lubricating fluid. The samples obtained were analyzed by Raman spectroscopy, showing spectra with parameters characteristic of DLC. FEG-SEM images of the samples showed that the deposited film broke into flakes ranging from hundreds of nm to tens of µm. Tests were carried out to determine the coefficient of friction (COF) between steel-steel pairs and DLC-coated steel pairs with and without lubrication, with a normal force of 10 N, for 24 hours. The lubricant mixtures showed a COF reduction compared to pure mineral oil, and the addition of DLC particles to this mixture resulted in a significant COF reduction. The mixture of DLC flakes in mineral oil/vacuum pump oil produced values similar to the DLC against DLC pair, with direct deposition, allowing low friction regimes even in systems that do not allow direct deposition.

Acknowledgment: Capes, ANP/Petrobras, CNPQ / INCT de Engenharia de Superfícies

References:

[1] J. Robertson - Materials Science Engineering R-Reports - 37(2002), 129

[2] T. Tokoroyama, et.al. - 2013 International Symposium on Micro-NanoMechatronics and Human Science (MHS), Japan

Influence of gas composition on carbon nanotubes-urea functionalization by plasma

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Carbon nanotubes (CNT) have been widely used in recent engineering applications due to their excellent mechanical properties, ease of surface modification, and capability of high adsorption level, which makes them able to be used, for example, as an adsorbent agent in water treatment [1]. However, CNT must be functionalized before use to prevent agglomeration and improve chemical interaction. Plasma treatment is a green alternative to the wet chemical process that attends to that need once it is fast and free of solvent process that can provoke CNT surface modification. In the present work, CNT was mixed with urea in the solid state and then treated in 35 W radio frequency plasma, in 1 Torr, for 30 minutes, following the instructions of another work [2]. Three combinations were set to evaluate the gas composition influence on plasma treatment of CNT: treatment with Ar, Ar, and O2, or Ar and N2. Both gas mixtures were done with a partial pressure of 80 % Ar. After treatment, all samples were methanol washed, intending to remove the possible chemically unreacted urea. Characterizations were conducted by Fourier Transformed Infrared (FTIR), Thermogravimetric analysis (TGA), Brunauer-Emmett-Teller (BET) adsorption method, and X-ray Photoelectron Spectroscopy (XPS). The oxygen concentration decreases with the change in the gas composition, and the nitrogen content on the NTC surface is relatively low, independent of the gas composition, with the highest value for sample NTC+Urea_Ar+N2 (0,35 %at.).

Acknowledgement

The authors would like to thank CAPES, CNPq and FAPESC by the financial support to the research, and to the LabPasma and Multi-User Facility infrastructure from UDESC/CCT.

References:

[1] R.B.Onyancha et al., Journal of Molecular Structure vol. 1238, 130462 (2021).

[2] T.T.Steffen et al., Applied Surface Science vol. 583, 152493 (2022).

In situ structural and mechanical analysis of the hydrogen-expanded austenite

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Austenitic stainless steel is widely accepted for hydrogen-related application equipments, owing from its low hydrogen embrittlement susceptibility [1]. Here, the first stages of hydrogenation were analysed and compared with the well-known N-expanded austenite. A 4-hour H-cathodic charging at 0.1 A/cm² in 1M H_2SO_4 electrolyte applied to a superaustenitic stainless steel produced a metastable H-expanded phase by interstitial solid solution. The in-situ XRD analysis disclosed two distinct lattice expansions and the eventual return to the pristine FCC lattice in about 1-day time. The two expanded phases showed an strain inversion between crystallographic planes, which has been observed in nitrided austenitic steels [2]. The mechanical behaviour of the modified surface revealed a twofold hardness increase and elastic modulus 17% higher due to the H-inlet, both of them returning progressively to the pristine values while hydrogen diffused out from the material. At a constant indenter load test, the H-charged surface underwent higher indenter penetration during the creep stage of the test. This is consistent with a competitive effect, where the solid solution strengthening overcomes the H-enhanced plasticity in the initial period of hydrogen outgassing.

Modification in photodegradation pattern of polypropylene by coating with PECVD organosiliconthin films

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Polypropylene (PP) is a material widely used in the manufacturing of parts for external use, however, this material suffers degradation when exposed to light in the presence of atmospheric oxygen. The degradation occurs by a series of oxidative reactions, which generate an increase in the crystalline domains on the surface, leading to crack formation. The cracks usually follow a parallel disposition pattern and are many micrometers in , these cracks make the material more brittle. Therefore, there is a search for ways to protect the PP from weathering, such as painting the surface of the material. However, the paint layer, despite protecting the material, promotes an aesthetic modification in the parts, which is not always desired. Thus, plasma coating can be a solution to protect PP from photodegradation. The samples were pre-treated with oxygen gas plasma for 5 min at 50W power. Organosilicone thin films were deposited by Plasma Enhanced Chemical Vapor Deposition, for 15, 30, 45 and 60 min, in an atmosphere with a 50:50 mixture of hexamethyldisiloxane and oxygen, with power of 80 W. The produced sample set was exposed to UV-C light in a photodegradation chamber, for the interval of 196 hours. The degradation of the samples was followed by Scanning Electron Microscopy and Fourier Transform Infrared Spectroscopy. The results suggest that the thin film promotes a mechanical anchorage that reduces the dimensions of the cracks formed by photodegradation in the coated PP relative to the uncoated material. This phenomenon occurred in all samples, regardless of the thickness of the films. However, they did not offer photoprotection to the polymer, this was evidenced by the formation of hydroxyl and carbonyl chemical groups, characteristic of photodegradation of the polymeric substrate.

Acknowledgments:This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Obtaining Vibrational and Rotational Temperatures in Plasma Microjets by Optical Emission Spectroscopy.

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Electrical discharges operating at atmospheric pressure and open environment, call attention due to the great possibility of application, ranging from superficial modifications of materials to biomedical and agriculture applications. Determining the operating temperatures of the plasma jet is important to ensure, among other things, that the plasma jet operates in a way that does not cause unwanted effects when applied [1,2]. MassiveOES is a software developed specifically for the analysis of optical emission spectra of plasma discharges, its objective is to focus on the experimental and scientific challenges that you will encounter during plasma spectroscopy. It is developed in python library by researchers Dr. Jan Vorac and Dr. Petr Synek from Masaryk University in Brno, CEPLANT center, and one of his goals is to obtain rotational and vibrational temperatures from the deconvolution of experimental curves of the emission spectrum of diatomic molecules. The objective of this work was to study the optical emission spectra of argon and helium plasma microjets to determine their vibrational and rotational temperature, using the MASSIVEOS software. It was possible to observe that the rotational temperatures of helium microjets ranged from 320 K to 430 K, thus showing that these kind of microjets can be applied in surface modification of materials of the most diverse natures and characteristics, as well as in medicine and agriculture.

Physical and mechanical properties evaluation ofWC-10 wt.% Co with incorporations of carbon nanotubes via Spark Plasma Sintering

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The combination of the high hardness of the tungsten carbide with the fracture toughness of the cobalt binder makes the WC-Co composite an important material for the tooling industry. However, the incorporation of structures in order to improve certain properties of this class of materials is the subject of several researches. One of these structures are the carbon nanotubes (CNTs), which are incorporated in several ceramics, alloys, metals, because of their unique properties. This work aimed to evaluate the physical and mechanical properties of WC-Co with the addition of carbon nanotubes, in the amounts of 0.10, 0.25, 0.50 and 1.00 wt.%. As sintering methodology, the Spark Plasma Sintering (SPS) technique was employed. The sintering process occurred under temperature of 1200 ℃ and pressure of 40 MPa, during a period of 5 minutes. The physical and mechanical properties of the studied systems were evaluated in detail, observing the increase of the apparent density, the stability of the Vickers hardness and the increase of the Young’s modulus of the system with 0.10wt.% CNTs - compared to the system without CNTs incorporated - reaching averages of 14.27 g/cm3, 14.96 GPa e 451 GPa, respectively. The fracture toughness increased gradually up to the system with 0.50wt.% CNTs, reaching the value of 12.93 MPa.m1/2.The values of compressive strength and splitting tensile strength also increased progressively to the system with 0.25wt.% CNTs, reaching the averages of 2905.77 e 474.30 MPa, respectively. Considering the results obtained by the characterization techniques, the system with the incorporation of 0.10wt.% CNTs presented the best performance. For the systems with the highest amounts of CNTs, the decrease in the values of the evaluated properties occurred due to the agglomeration of the carbon nanotubes.

Plasma treatment to improve hydrophobicity ofbacterial cellulose.

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Bacterial cellulose (BC) has attracted attention in recent years, since its easy synthesis and green appeal make it a promising material to replace animal leather in the fashion and textile industry. However, an inherent issue related to BC regards its wettability (contact angle of about 45°). The hydrophilic nature of BC means it is not suitable for wear in conditions where there may be increased humidity, such as next to the human skin, and therefore, cannot be subjected to domestic washing [1]. Plasma treatment is a sustainable alternative to overcome that problem, once is capable to coat the BC surface with hydrophobic films formed through carbon gases [2]. In the present work, bacterial cellulose was synthed using kombucha, following the instructions of another work [3], and subsequently treated by DC pulsed plasma. In the way to evaluate the plasma parameters on the film formation above de BC, four different combinations were set, changing gas compositions (Ar and C_2H_2; or Ar, C_2H_2 and H_2), time (5 or 30 minutes) and polarity time off (2 or 4 µs). Characterizations were conducted by Fourier Transformed Infrared (FTIR), X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscopy with Field Emission (SEM-FEG), and Water Contact Angle. For all the treatments, results indicate a carbon film formation on the surface of bacterial cellulose, resulting in an improvement of material hydrophobicity. The best plasma condition (Ar and C_2H_2, 30 minutes, 4 µs of polarity time off) was defined by the highest water contact angle obtained: 102°, in comparison to 33° forthe non-treated BC.

Acknowledgement
The authors would like to thank LabPasma and Multi-User Facility infrastructure from Santa Catarina State University's Technological Sciences Center.

References
[1] J.Wood, Biomimetics vol. 4, p. 16 (2019).
[2] M.M.A.N.Ranjha et al., Trends in Food Science Technology vol. 134, p. 162-176 (2023).
[3] B.L.Lima et al., MIX Sustentável vol. 7, p. 153-164 (2021).

Preparation of boron doped DLC film via enhanced-PECVD process

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Plasma Enhance Chemical Vapor Deposition (PECVD) is widely used to the growth of Diamond-Like Carbon (DLC) films on surfaces with large areas and irregular geometry. Due to its excellent tribological, mechanical, chemical, properties, etc DLC films are used as a protective coating. And due to great interest in applications technological and medical provide by boron, in this works, it is explored the incorporation of boron into the DLC films by using the enhanced PECVD process. The system was prepared with an additional cathode, placed inside the PECVD reactor, to confine the plasma, and a bubbler, placed outside the reactor, to produce the boron solution. Thus, the boron doped DLC film was grown on Ti-6Al-4V and Si substrates by varying the pressure and voltage. They were varied from 3mTorr to 7mTorr and from 0.7kV to 1.0kV, respectively. Changes in structural, mechanic, tribological and electric properties of the doped DLC film were evaluated. The results evidenced presence of boron in all doped samples. The electric resistance was strongly reduced in conditions of higher pressure and voltage. Our results showed that the pressure and voltage variation resulted in a progressive graphitization of the doped films. Other analyses are in progress.

Acknowledgements:
This work was support by Concelho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) grants # 102497/2022-2 and 106517/2023-6, and Fundação de Amparo à Pesquisado Estado de São Paulo (FAPESP), grant # 2017/08899-3 and 19/18572-7.

Production and characterization of $Al_xGa_{1-x}N$ films by reactive dual magnetron sputtering

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This study focuses on the production and characterization of $Al_xGa_{1-x}N$ films using reactive magnetron sputtering. The films were grown directly on Si (111) substrates using a 4-inch Ga target powered by a 600W RF power supply and a 1-inch Al target powered by a 1kW DC power supply. The liquid Ga target was positioned to directly face the rotating substrate holder at a distance of 90 mm, while the Al target was located at 150 mm from the center of the substrate holder and tilted at a 45-degree angle. A 1:2 Ar/N_2atmosphere was maintained at 5 mTorr, and the substrate temperature was kept at 550°C during the 120-minute deposition process. To ensure uniformity, the substrate was rotated at 6 rpm. Ten samples were grown using different combinations of individual target power for Ga and Al to adjust composition. The composition of the samples was analyzed using energy dispersive spectroscopy, and the thickness was measured using profilometry. X-ray diffraction (XRD) was used to study the crystalline structure of the samples. The deposition rate showed a strong dependency on the target powers, with a greater increase observed for Ga power compared to Al power. The target size and geometry of the deposition chamber, including the different distances and angles between the substrate holder, as well as the different power supply used (RF and DC) and the different sputtering yield of Al and liquid Ga, may explain this difference. The XRD results revealed that all samples exhibited only peaks related to the wurtzite phase, with a stronger c-axis orientation observed for the Al richer samples ($x > 0.24$), which were produced using 30 W on the Ga target. The lattice parameters $a$and $c$decreased with increasing x, with a deviation from linear behavior that could be attributed to the strain on the produced $Al_xGa_{1-x}N$ films.

Acknowledgments:
Fapesp [Grants 2015/06241-5], CNPq [Grants 428591/2018- 3, 159754/2018], CAPES [Grants 88887.509345/2020-00 and 88887.508992/2020-00].

Silver nanoparticles deposition on polyamide 6.6 fabrics by hybrid corona-dielectric barrier discharge asan alternative for increasing the effectiveness of face masks

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Silver nanoparticles (AgNPs) have been widely investigated due to their antimicrobial activity against several pathogenic microorganisms. This property makes them suitable for a wide range of applications in medical and healthcare products [1]. One promising use of these nanoparticles is their incorporation into textiles to enhance the effectiveness of face masks against microorganisms associated with respiratory tract infections. In this work, AgNPs were deposited on polyamide 6.6 fabrics by hybrid corona-dielectric barrier discharge plasma at atmospheric pressure with a plasma dosage of 1800 $W.min.m^{-2}$at 19 kHz. This process consists of two steps: surface activation in air plasma and AgNPs deposition using an aqueous dispersion of stabilized AgNPs, which is nebulized into the discharge region. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) analysis confirmed the AgNPs deposition on the textile surface, showing that plasma processing is an effective technique for AgNPs incorporation on polyamide 6.6 fabrics. Microbiological analysis based on the American Association of Textile Chemists and Colorists (AATCC) 100-2019 test method evaluated the antimicrobial effect of AgNPs on *Staphylococcus aureus*and *Klebsiella pneumoniae*, which are respiratory tract infection-related bacteria. For this analysis, bacterial suspensions were inoculated into untreated (without AgNPs) and treated (with AgNPs) samples. Significant decreases in bacterial counts were detected in textiles containing AgNPs, with reduction percentages up to 74.63% compared to the untreated sample, indicating that AgNPs are a potential alternative for increasing the effectiveness of face masks.

Acknowledgments: CNPq, CAPES (88887.509033/2020-00) and FAPESP.

References: [1] BRUNA, T. *et al.* Silver nanoparticles and their antibacterial applications. **International Journal of Molecular Sciences**, v. 22, n. 13, p. 7202, 2021.

$SiO_xC_yH_z$-TiO_2 Films Deposited in Low Pressure Plasma with Photocatalytic Activity under UV and Visible Radiation

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This study investigates the possibility of preparing of $SiO_xC_yH_z$-TiO_2 photocatalytic films in a single step plasma process that combines the plasma enhanced chemical vapor deposition (PECVD) of hexamethyldisiloxane and the reactive sputtering of TiO_2. The samples were prepared in a capacitively coupled reactor fed with hexamethyldisiloxane (HMDSO), O_2 and Ar, at a working pressure of 4.0 Pa. Plasma was ignited by application of radiofrequency signal (150 W, 13.56 MHz) to the lowermost electrode where titanium dioxide powder, P25, was accommodated. Substrates were attached to the uppermost grounded electrode and process time was of 3600 s. It was investigated the effect of the oxygen proportion in the plasma phase on the properties of the films. The results show the formation of organosilicon, silicon oxicarbides up to silicon oxides structures, all containing TiO_2 particulate. The concentration of granular structures increases with the proportion of O_2. Surface free energy decreases with O_2%. All films presented photocatalytic activity under ultraviolet and visible light irradiation but the highest efficiency was attained when irradiated with ultraviolet light (up to 60% of degradation for 90 min of irradiation). Authors thank to FAPESP (Process number 2020/06448-7 and 2017/21034-1) for finatial support.

Stamping with natural dyes and thickeners on plasma-treated fabrics

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Textile stamping plays one of the main and important roles for the textile chain, because it is one of the most demanding stages and that comes closest to art, where great value is added to the product, with this has been changes and several possibilities arise for process improvement. In the stamping are used thickeners for formulations of pastes that are colored and stamped. In the coloring are used synthetic dyes, which are aggressive to the environment. Thus, in this work we used natural dyes and thickeners that are less aggressive and from sustainable sources. In the preparation of pre-stamping tissues plasma was used which is a unique treatment, in which a large amount of water, chemical agents, energy and process time are consumed. The objective of this work was to stamp on tissues treated with plasma, using thickeners and natural dyes. Natural thickeners of sodium alginate, guar and tamarind were used for formulations of pastes, and colored with natural dyes of annatto and turmeric, then they were stamped on cotton fabrics that were previously submitted to treatment with oxygen plasma for 5 minutes, with a power of 100 W and pressure of 100 mTorr. Then the prints were fixed with steam at 100°C for 40 minutes. then the fabrics were washed with room temperature water to remove the unfixed dyes and thickeners. The results showed that the prints with natural dyes in plasma fabrics were similar to those of conventionally treated fabrics. It was also observed that the natural thickeners had good performance in the stamping, and the sodium alginate thickener, had better results in the sense of ease of removal in subsequent washes. As a product we can get printed fabrics using sustainable sources in addition to using plasma in the pre-stamping preparation, which makes the whole process beneficial to the environment.

Structural changes produced by He plasma treatmenton Hydrogenated Carbon Nanoparticles

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In this work, the structural changes caused by He plasma treatment of amorphous hydrogenated carbon nanoparticles were studied. The nanoparticles were produced by RF-dusty plasmas with acetylene as a precursor. The particles were dispersed in acetone and dripped on a silicon substrate, then treated with an RF capacitively-coupled glow discharge plasma in 50 mTorr He atmosphere, with -700V self-bias applied to the substrate As observed by FEG-SEM, before the treatment, the nanoparticles formed large aggregates over all the silicon, afterward, some particles were sputtered and the remaining ones fused with neighboring particles. Raman Spectroscopy of the sample before and post-treatment revealed a drastic difference in the observed spectra. The pristine carbon nanoparticle spectra displayed only a broad photoluminescent(PL) spectrum. After the treatment, a reduction of the PL spectrum was observed, showing the D and G band peaks at 1326 and 1527 cm^{-1}respectively, with G-band FWHM of 130 $cm^{-1}$and I(D)/I(G) of 0,65. We concluded that the PL decrease was due to the creation of larger sp^2clusters and sp^3defects on the hydrogenated carbon matrix, because of the non-equilibrium effects during ion bombardment, which can be explained by the knock-on subplantation model. The observed features of the Raman spectrum were strongly different from those obtained from samples thermally treated at temperatures ranging from 200°C to 500°C.

Acknowledgments:
Faperj, CNPq, CAPES, INES(INCT)

References:

[1]J.Robertson, Diamond-like amorphous carbon. Materials science and engineering: R: Reports 37(4-6), 129-281 (2002)

[2]Rusli, J. Robertson, and G. A. J. Amaratunga. Photoluminescence behavior of hydrogenated amorphous carbon. Journal of Applied Physics 80.5, 2998-3003 (1996).

Study of the Properties of Carbon Steel Coated with Organosilicon Film Containing Cerium Deposited by PECVD

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Although carbon steel is a widely used material, it is easily oxidized when exposed to the atmosphere. An interesting group of protective layers are the so-called smart coatings, which provide a viable solution to deal with the challenges of corrosion in a wide range of applications due to their promising properties to regenerate after suffering some damage, which is known as self-healing. Among these coatings are those containing cerium. In view of this, the present work aims to investigate the effectiveness of the dilution of cerium nitrate in TEOS (tetraethyl orthosilicate) for the deposition of protective organosilicon films against corrosion on carbon steel by the technique of Plasma Enhanced Chemical Vapor Deposition, PECVD. Plasma was generated by applying a DC voltage (400 V) in a vapor environment of cerium nitrate diluted in TEOS solution and argon. The variation of the total pressure of the gases in the properties of the layers was investigated. Scanning electron microscopy (SEM) was used to evaluate the surface microstructure of the layers. A dispersive energy analyzer coupled to the microscope was used to identify the chemical species present on the surface. Infrared spectroscopy (FTIR) was used to analyze the chemical composition and molecular structure of the layers. Electrochemical Impedance Spectroscopy (EIS) was applied to evaluate corrosion resistance. The thickness and deposition rate of the films were determined by profilometry, while the wettability was determined by the sessile drop method. The infrared spectra revealed groups of organic species and also bands referring to the asymmetric stretching of the nitrate ion, which comes from the dissociation of the $Ce(NO3)3$ salt.

Acknowledgements:

The authors thanks to Fapesp (2020/06448-7) (2017/21034-1) (2013/050012-7) and to ABC Federal University for financial support.

Sulfate Radical SO4•— Activation by Plasma Electrolysis studied by Computational Chemical Dynamics

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In situ generation of either hydrated electrons (e_{aq}^-) or radicals such as sulfate ($SO_4^{\cdot -}$) and hydrogen (H·) have a central role for Advanced Oxidation Processes (AOPs) of enviromental pollutants like perfluorooctanoates (PFOAs) and perfluorooctanesulfonates (PFOS), being the plasma electrochemistry an electrified technology capable of producing all those three species in any water matrix. Making maximum use of the recently developed model for the chemical dynamics driven by the plasma-induced hydrated electrons (e_{aq}^-) beneath the electrified plasma-liquid interface (EPLI), this work elucidates the employment of plasma electrolysis to reduce $S_2O_8^{2-}$from an empirical theoretical perspective. This work proposes chemical reaction networks driven by the radical $SO4^{\cdot -}$and analyzes their respective chemical dynamics via computational experiments. In summary, this work identifies the core mehcanism of sulfate radical activation via plsama electrolysis in two water matrixes, namelly (a) acidified ultrapure water and (b) chloride-rich water. The results demonstrate that plasma electrolysis enables SO4·— local concentration as high as 350×10^{-6}mol L^{-1}, which can lead to the instantaneous mineralization (full oxidation) of up to 1.18 ppm of phenol, used here as a model pollutant. Besides identifing the core mechanism of sulfate radical activation by the plasma electrolysis, the results of this work offers valuable assistance in order to carry out bench experiments of PFOAs and PFOS oxidation.

Surface properties of Nb_2O_5/AZO thin films grown by HiPIMS at room temperature

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Perovskite solar cells (PSCs) have shown remarkable progress in recent years with rapid increases in conversion efficiency, from reports of about 3% in 2006 to over 25% today [1]. Transparent and selective electrodes are crucial for PSCs performance. In particular, niobium pentoxide (Nb_2O_5) is a promising material to be used as electron selective layer due to its high stability [2]. Additionally, Nb_2O_5 is non-toxic and derives from readily inexpensive source materials. In this work, Nb_2O_5 and Nb_2O_5/AZO thin films were grown on glass at room temperature. A 1kHz High-power impulse magnetron sputtering (HiPIMS [3]) source at a power of -600 V on pulse and different duty-cycle was used. Optical reflection and transmittance were measured using a UV-Vis-NIR spectrometer (Lambda 750, Perkin Elmer) over the wavelengths from 190 nm to 2500 nm. All samples show average transmittances greater than 80% in the visible region. The refractive index at 500 nm of Nb_2O_5 and AZO thin films was around 2.4 and 1.9, respectively. Surface morphology was measured with atomic force microscopy (XE-100, Park Instruments) operating in air. All AFM images of the films were analyzed using scaling concepts. The RMS roughness remained around 10 nm for AZO (650 nm thick) and Nb_2O_5 (75 nm)/AZO (650 nm) The wettability behavior was measured using a Goniometer (DSA25E, Kruss). AZO and Nb_2O_5/AZO surface shows a contact angle lower than 75 and 50°, respectively.

Acknowledgements:
CAPES, CNPq, FAPESP (Process number: 17/15853-0) and CBMM Company for niobium metallic target.

References:
[1] X. Jiang, Z. Zang, Y. Zhou, H. Li, Q. Wei, and Z. Ning, Accounts of Materials Research 2, 210 (2021).
[2] S. L. Fernandes, L. G. S. Albano, L. J. Affonço, J. H. D. Silva, E. Longo, and C. F. O. Graeff, Frontiers in Chemistry 7, 1 (2019).
[3] A. Anders, Journal of Applied Physics 121, 171101 (2017).

The influence of exposure time of a non-thermal plasma jet prototype applied to biomedical pathogensfor clinical use

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The growing microbial resistance to different antimicrobial agents has been a global concern and factors such as excessive and improper use of antibiotics have been pointed out [1]. Thus, the search for new effective treatment methods with a broad antimicrobial spectrum is urgent [2] and non-thermal plasmas have stood out for their antibacterial and antifungal effects [2;3]. This study aimed to evaluate the antimicrobial action of a home-made prototype of cold atmospheric plasma jets (CAPPJ) against pathogenic species. Suspension of Gram-positive bacteria ***Staphyloccocus aureus***and Gram-negative P*seudomonas aeruginosa*and *Candida albicans*fungus were standardized with sterile saline solution (10^6cell/mL). Inhibition tests were made on Brain Heart Infusion agar plate for bacteria and Sabouraud dextrose for fungus that were seeded with standardized suspensions followed by drying for 15 min. The antimicrobial effect of CAPPJ with helium (He) gas was evaluated at a power of 75 mW, distance of 2 cm and at times of 1, 2, 3 and 5 min. Control group was exposed to He gas only (no plasma). The tests were performed in triplicate at two different times. The inhibition zone’ s area was calculated using the Kruskal Wallis statistical test followed by the Dunn''s multiple comparison test. An increase in the zone of inhibition area was observed as the time of exposure to CAPPJ increased in all evaluated microorganisms. Control group did not show zone of inhibition. For ***S. aureus***and ***P. aeruginosa***bacteria, a statistically significant difference was observed between the area of inhibition after 3 min (2.74 mm^2, p=0.0008; 32.97 mm^2, p=0.0015) and 5 min (6.41 mm^2, p=0.0004; 40.68 mm^2, p=0.0008) compared to control. Differently, the zone of fungal inhibition was statistically different from control group after 2 min (2.74 mm^2, p=0.0354) and 5 min (7.58 mm^2, p=0.0001). These findings demonstrate the promising antimicrobial capability of the He CAPPJ for future biomedical applications.

Thermo-optical and mechanical properties of HastelloyX superalloy nitrided surfaces.

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The nickel-based Hastelloy X superalloy was nitrided by plasma imersion ion implatation for the first time in this study. The acceleration voltages were 8 kV and 10 kV, resulting in 360 ºC and 330 ºC, respectively. Crystal structure and surface mechanical properties were compared with the respective substrates, aged by the process heating. The modified layers retained up to 20% of N atoms in both conditions. The nitrogen diffusion in the matrix obeyed a complex and dynamic correlation with temperature due to the competitive nitrogen retention in interstices and precipitates. Raman spectroscopy allowed analyzing the formation of nitrides as Cr2O3, Fe2O3, Fe3O4, FeCo3 and TiN on the top surface (few nanometers). The structure at deeper regions, as inferred by X-ray diffraction, comprised the N-expanded austenite phase (the S-phase) as the main modification imposed by nitriding. The nitrogen inlet yielded strengthened cases, with hardness values reaching 8 GPa and elastic modulus 200 GPa (4 times and 40% higher than the substrates, the values were 3,8 GPa and 160 GPa, respectively). By photoacoustic spectroscopy, band gap values are in the range of the u-v energies for 8 and 10 kV treatments, respectively. These results are of intrinsic importance for the load bearing capacity and heating dissipation of the modified surfaces on the nickel superalloy.

Titanium protective, self-cleaning thin films depositedby plasma magnetron sputterring on the surface of solar panels installed on a floating solar plant

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Solar panels are currently a clean form of energy generation and are gradually replacing the energy matrix of countries. Centralized generation usually takes up large spaces that could be used for agricultural or reforestation activities. Therefore, their implementation in hydroelectric power plant lakes is an alternative. The exposure of these panels is conditioned by the accumulation of surface contaminants that can be of organic and/or inorganic origin. This new surface limits the passage of light, reducing the capacity for electricity generation. Periodic manual or mechanized cleaning methods are employed to remove the cemented dirt on the surface, however, this involves the use of qualified professionals, water resources that may not be available in many cases, cleaning chemicals, etc. With the aim of mitigating and maintaining the surface of the panels clean for longer, this research investigated materials and processes for the deposition of thin titanium films on the surface of glass sheets using the magnetron sputtering plasma technique. The films obtained were deposited on glass sheet surfaces with thicknesses varying linearly with the deposition time. After the heat treatment, the transmittance of the coatings with times up to 30 s was approximately 98%. From the wettability test, the hydrophilicity of the new surface was observed, with wetting angles below 15°, being considered self-cleaning.

Acknowledgements:

The authors would like to acknowledge the support in financial resources and infrastructure by the companies ELEJOR S. A., ANEEL and Lactec, R D project 2945-1000/2017; CNPq process DT 1C, 30.8777/2020-4; CNPq Law 8010/90, DI 18/1041222-8, LI 18/1680554-2; and DI 19/0528208-9; LI 19/0641121-7; and Pós-graduates program UFPR-PIPE, Lactec/IEP, UEPG/DEMA and CLabmu, UEPG laboratories.

Titanium surface modified by oxide films containing calcium and apatite to improve bone integration

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Calcium-containing coatings, such as apatite, are important for bone integration of implants because the calcium release triggers a series of biological responses that favor a stable chemical bond. It is known that the direct deposition of apatite on titanium can lead to adhesion failures, but the effectiveness of adhesion can be improved by structural and morphological modification of the surface of the material. This study evaluated the influence of titanium oxide containing calcium as an intermediate layer. This layer was prepared by two different methods: sol-gel/dip coating and plasma-assisted oxidation (PEO), on which the apatite film was deposited via the sol-gel method associated with dip-coating. The intermediate films consisted of rutile and anatase TiO2, and CaTiO3. The apatite film was composed of two phases: hydroxyapatite (HAp) and β-tricalcium phosphate (β-TCP). Roughness analyzed by Atomic Force Microscopy (AFM) decreased when apatite was deposited on the intermediate films, while the wettability increased. Micrographs taken byScanning Electron Microscopy (SEM) showed that the modified surface exhibits cracks andsplits when prepared via sol-gel, while PEO processing produced a porous surface. Thethickness of apatite films deposited directly on Ti-c.p. (commercially pure titanium) was 1.5µm, but varied to 0.6 µm and 2.5 µm when deposited on CaTiO3 intermediate films obtainedvia sol-gel and modified by PEO, respectively. Both coatings reduced the corrosion rate, aswell as no significant changes were observed for bending stress and elasticity modulus. Inthe direct contact toxicity assay, pre-osteoblast viability (MC3T3, subclone 4) was lower onsurfaces modified via PEO, apatite, and CaTiO3 with apatite. On the other hand, the deposition of apatite films on PEO-modified Ti-c.p. significantly increased viability. The adhesion of pre-osteoblasts was higher on all samples compared to the control.

Treatment of commercial fabric with a Conical-Shaped Atmospheric Pressure Plasma Jet

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The textile industry is one of the largest and most developed industries in the world, requiring constant updates and modernizations in various processes [1]. One of the common and widespread practices in the industry is starching fabrics [2], which improves their resistance to friction and prevents unraveling of the textile structure. However, starch coating reduces the wettability of the textile fiber, making it difficult for subsequent finishing processes, such as painting. Typically, after the coating process, fabrics undergo further treatment to improve their wettability, aiding in the finishing processes of the textile production line [3]. The study aimed to investigate the effect of atmospheric plasma generated by a conical-shaped atmospheric pressure plasma jet. Cotton (CO) and polyester (PES), the two most commonly used textile materials, were used to create fibers, with both fabrics constructed using the Oxford method and distinct compositions. The first fabric was made entirely of cotton, and the second was a blend of 33% cotton and 67% polyester. To analyze the physical and chemical changes caused by the treatment, several surface characterization methods were used, including the vertical wicking test, Fourier-transform infrared spectroscopy (ATR-FTIR) with attenuated total reflection, scanning electron microscopy (SEM), and X-ray photoelectron spectroscopy (XPS).

References:

[1] P. Frizoni, “PROCESSO ECOLÓGICO PARA O TINGIMENTO DO ALGODÃO purga ezimática”, TCC, Americana, 2011.
[2] L. Beltrame, “Caracterização de Efluente Têxtil e Proposta de Tratamento”, 2000.
[3] C. P. de R. H. CPRH, “ROTEIRO COMPLEMENTAR DE LICENCIAMENTO E FISCALIZAÇÃO PARA A TIPOLOGIA TÊXTIL”, Recife, 2001, p. 1-10.

Zinc Oxide Film on Glass Substrates by Means of Plasma Immersion Ion Implantation and Deposition

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Zinc Oxide, a semiconductor material, presents many interesting characteristics to electronics industry[1-6]. This paper shows a process for coating glass with ZnO by Plasma Immersion Ion Implantation and Deposition (PIII&D). The process consists of zinc deposition on the glass surface and post-oxidation to form ZnO. In the process, small pieces of metallic zinc filling the top of a crucible are immersed in an argon and oxygen plasma; the crucible is positively biased and draw electrons from a termionic electron emitter, which is composed of a tantalum foil covered with (Ba, Sr, Ca) O. The low work function of this oxide cathode is essential to supply enough amount of electrons to heat the crucible to temperatures ranging from 400 to 450 o C, leading to vaporization of the zinc. The zinc vapor is partially ionized by of collisions with argon ions and electrons from the plasma. High negative and repetitive voltage pulses (10kV/20μs/250Hz) applied to a sample holder containing the glass slices causes the implantation of zinc ions into the glass surface; between pulses zinc is deposited onto glass substrate. After treatment, the samples were characterized by X-ray diffraction (XRD) in the mode of thin films, atomic force microscopy (AFM), scanning electron microscopy (SEM), Optical Profilometry and Transmittance. The XRD analysis showed the presence of crystalline ZnO after annealing. The AFM analysis as deposited showed the formation of microstructures. Topography viewed by SEM and Optical Profilometry showed the formation of structures with nanostructures after annealing. The optical transmittance reached 51.8% of transmittance in visible wavelength range. The PIII&D allowed to growing nanostructures of ZnO on glass without the need of a buffer layer.

P-Polysaccharide-based Materials

Expanding the property space of polysaccharides through peptide conjugation

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Polysaccharides combined with peptides are ubiquitously found in biology and fulfil destinctive structural and signalling functions. The best known examples include peptido- and proteoglycans, both of which have very different (supra-)molecular forms and biological roles. Imitating those structures by semi-synthetically combining polysaccharides with short peptides can potentially contribute to medical therapies including drug delivery or regenerative medicine. Drug delivery requires the binding and controlled release of drugs by splitting covalent bonds or non-covalent complexes, whereas regenerative medicine often makes use of cross-linkable but degradable gels to embed cells or tissue. Derivatizing polysaccharides with peptides in a very defined way requires special chemical tools, not all of which are yet availble. Combining biological methods with chemical ones is one of the options to create new conjugates. Upon attachment of peptides to polysaccharides, their structure and properties change significantly, requireing sophisticated analytical methods to study these. This presentation first gives an overview on the motivation and the state-of-the-art of studying polysaccharide peptide conjugates. It then communicates selected examples of where and how conjugates have been designed, analyzed and applied and concludes with suggestions for further studies of the topic.

Exploiting the potential of polysaccharide in the development of advanced bioinks for 3D bioprinting applications

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3D bioprinting is a technique with undeniable potential in the biomedical field, resulting in the fabrication of living structure analogs that may be used in tissue engineering, new drugs testing and diseases research. The cell-laden materials used to create these 3D structures are called "bioinks". The precise layer-by-layer deposition of the bioinks, in a computer-controlled manner, originates complex 3D constructs with tailored size and structure, loaded with living cells that could proliferate in the matrix. Understandably, the characteristics of the bioinks majorly influence the success of the 3D bioprinting procedure, and the performance of the resulting constructs. A perfect bioink should possess adequate rheological, chemical, mechanical, and biological features to originate stable structures where the living cells can thrive. Given so, the development of bioinks with improved properties is a very relevant and active research topic in this area. Particularly, hydrogel bioinks derived from biopolymers, especially polysaccharides, have been widely explored because of their low cytotoxicity, high hydrophilicity, and closely mimic the native extracellular matrix. Furthermore, the tunability and facile crosslinking of polysaccharides, together with their recognized abundance, place polysaccharides hydrogels in the spotlight regarding the design of novel bioinks [1].

In this talk, innovative studies about polysaccharide-based hydrogel bioinks for 3D bioprinting of diverse cell lines, will be presented and discussed, with emphasis on their compositions, rheological properties, printability, biological performance and role of polysaccharides in the bioink formulations.

Acknowledgments: Project CICECO—Aveiro Institute of Materials (UIDB/50011/2020, UIDP/50011/2020 LA/P/0006/2020) and project I D "NANOBIOINKS-CENTRO-01-0145-FEDER-031289

References:[1] M.C. Teixeira, N. Lameirinhas, J. Carvalho, A. Silvestre, C. Vilela and C. Freire, Int. J. Mol. Sci., 23, 6564 (2022)

Exploiting the properties of chitosans innanostructured films

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Chitosans are now used in two main types of nanostructured films, namely on layer-by-layer (LbL) films as matrix for sensors and biosensors and on Langmuir monolayers that mimic cell membranes. In this lecture, chitosan-containing LbL films will discussed which are used in preserving the activity of biomolecules in biosensors and as sensing materials in electronic tongues (e-tongues). For example, an e-tongue was built with LbL films made of chitosan, chondroitin sulfate, sericin and gold nanoparticles to detect S.aureus in milk, which is used in the diagnosis of mastitis. A precise diagnosis was achieved by treating the impedance data obtained with the e-tongue a machine learning method. As for the chitosan incorporation into Langmuir monolayers, a set of results will be employed to correlate the activity of chitosans as bactericide agent and their interaction with cell membrane models. Of particular relevance will be the discussion of tailored chitosans synthesized in order to be soluble in water, which permits a more realistic comparison of biological effects.

FUTURE POLYSACCHARIDES RESEARCH AND DEVELOPMENT TRENDS PRESENTED IN THE EPNOE ROADMAP 2040

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EPNOE was established in May 2005 as a European Polysaccharides Network of Excellence Network with funding from the European Commission (EC) and was registered as a non-profit association and continued its activities after the end of EC funding in October 2009. EPNOE prepared and presented in the spring 2023 the EPNOE Roadmap 2040 (https://www.epnoe.eu/discover/research-roadmap/), which has strong and synergistic links to EU strategies and objectives, including the EU Bioeconomy strategy, EU Green Deal, EU Missions, Horizon Europe Clusters, and EU Industrial Strategy. The EPNOE Roadmap 2040 strategic areas are prepared considering the broadness of the topic and the gaps and uncertainties of available literature. The roadmap is based on the knowledge and skills of experienced researchers, scientists and academics. Each topic discusses the current challenges, expected achievements and developments in the coming 20 years, and the required research activities needed for the understanding of the most important scientific and societal questions to be answered. The main goal of this Roadmap is to arrive at solutions for a sustainable use of biomass resources while keeping biodiversity. The strategic choice of research activities in polysaccharides as a part of a very diverse biomass is very important and essential in achieving a sustainable development of our societies.

MATERIALS RECENTLY DEVELOPED AT THE GMPC- UEM / LIMAV-UFPI BASED ON POLYSACCHARIDES AND THEIR POTENTIAL FOR TECHNOLOGICAL USES

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This lecture will present recent results (last 10 years) from our research groups at GMPC/UEM (Polymeric Composite Materials Group, at UEM-Maringa-Brazil); and at LIMAV/UFPI (Advanced Materials Interdisciplinary Laboratory, at UFPI-Teresina-Brazil), in developing materials based on polysaccharides with potential for technological applications in the following fields: i) environmental (adsorption of contaminants using hydrogels); ii) agriculture (soil conditioners for humidity maintenance and carriers for releasing of plant nutrients); and iii) biomedical (controlled release of drug bactericidal actio substrates for cell growth; materials with virucidal action to combat the SARS-Cov 2 virus, related to the recent corona virus pandemic). The polysaccharidic-based materials are mainly prepared by means of three-dimensional matrices (hydrogels) polyelectrolyte complexation between anionic/cationic polymers of chemically modified (or not) polysaccharides in presence (or absence) of ionic liquid and by means of the electrospinning technique [1,2]. Several methodologies will be shown and discussed, targeting to obtain matrices of different geometries (cylindrical, spherical, irregular particles, thin films, nanofibers, etc.) of different sizes (macro, micro and nanometric scales). In the last part of the talk, information of the INCT-Polysaccharides, recently approved by CNPq-MCTI-Brazil, evolving research groups from 15 Brazilian universities, and at this moment in implementation, will be given.

Acknowledgments:FAPESPI, Capes and CNPq (Grant #406973/2022-9, #408767/2021-9 and 307429/2018-0). References: [1] - Pereira, AG Nunes, C Rubira, AF; Fajardo, AR. Effect of chitin nanowhiskers on mechanical and swelling properties of Gum Arabic hydrogels nanocomposites, Carbohydrate Polymers, 266, 118116 (2021). [2] - Spagnol, C; Fragal, EH; Pereira, AG Nakamura, CV; Muniz, EC; Follmann, HDM ; Silva, R; Rubira, AF. Journal of Colloid and Interface Science, 351; 705-715 (2018).

Mesoporous polysaccharide-based carriers as future sustainable materials for pharmaceutical applications

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Polysaccharides are abundant renewable materials with outstanding structural diversity and functionality. Polysaccharides have been extensively utilized in pharmaceutical applications such as thickeners, suspending agents, emulsifiers, stabilizers, gel forming agents, binders, disintegrating agents, matrix formers, release retardants, film formers, coating materials and mucoadhesive agents. They have been also exploited as excipient in different pharmaceutical dosage forms for example microparticles, suspensions, emulsions, gels, tablets, capsules, beads, microparticles, nanoparticles, liposomes, transdermal formulations, to name a few. In this talk we will present and discuss the design and fabrication of stimuli-responsive porous polysaccharide materials with dual function: drug amorphization and stimuli-responsive controlled delivery. Cellulose microspheres fabricated using water-based solvents and antisolvents were post-functionalized via oxidation or via oxidation and addition of amino acids. Hydrophobic drugs were encapsulated in mesopores of the microspheres resulting in amorphous solid dispersions. Drug-loaded microspheres showed pH stimuli responsive delivery at different pHs, simulating gastrointestinal conditions. The pH-responsive oxidized cellulose beads with good biocompatibility, low cost, and adjustable release rate have shown great potential in the field of controlled drug release. This is the first study that reports on exploiting soluble, porous, dialdehyde cellulose beads and novel pH responsive amino cellulose beads, showing excellent potential as a carrier for improving the rate and extent of dissolution of poorly soluble drugs and maintaining supersaturation.

Nanostructured biomass for multifunctional materials

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A wide range of biomass-derived polysaccharides has been widely explored by the scientific community lately in response to the run towards eco-efficient materials delivering advanced functionalities. Our group has been focusing on cellulose, chitin, pectin, starch, alginic acid and their derivatives, to mention a few, either as colloidal or molecular building blocks for (nano)composite films, coatings, mats, and parts with suitable properties for a range of applications, such as electronic devices, biocidal surfaces, delivery platforms, biodegradable packaging, edible films, and cell growth scaffolds. This talk will cover the most relevant aspects of the compositions, assemblies, and processing methods of such materials.

Acknowlegdements: FAPESP proc. 18/22214-6

3D printed bone scaffolds based on potato starch activated by hydroxyapatite nanoparticles replaced bySr2+

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3D printing is enables to the production of custom bone scaffolds and the use of starch (a natural polymer biocompatible and biodegradable) [1] incorporated by hydroxyapatite nanoparticles (HA) replaced by strontium ions is one potential biomaterial to induce bone formation and to reduce bone reabsorption [2]. In this context, this work evaluated the bioactivity of 3D printed produced bone scaffolds based on potato starch gels added with HA nanoparticles replaced by different molar percentages of Sr2+ ions (0,30,50,70, and 100%, HA_0, HA_30, HA_50, HA_70, HA_100) for a possible application in guided bone regeneration. The formulations (inks) were based on potato starchy hydrogels (10% w/w, d.b.) added of HA (5% w/w starch, d.b.). The scaffolds were printed using a 3D printing extrusion (BioedPrinterV4, Brazil). The printed scaffolds were characterized concerning swelling, biodegradability, cytotoxicity by MTT assay using MC3T3-E1 pre-osteoblasts cells, and the alizarin red assay to evaluate the ability of the membranes to induce mineralization. The HAp_100 showed the highest biodegradability value (70% in 14 days). The scaffolds showed significantly swelling in 2 h in medium (~60%); after this period, the swelling was not noticed. All scaffolds showed no cell toxicity. HAp_100 stood out positively in cell proliferation. Furthermore, scaffolds containing HAp had higher mineralization than ones without in both time intervals, and this effect was more evident with an increasing degree of Sr2+ substitution in the nanoparticles. It is evident that the incorporation of HAp and various levels of Sr2+ substitution has improved the properties of scaffolds for use in bone regeneration. Finally, 3D printing technology and starch holds significant promise for this application.

Acknowledgments:
FAPESP(2020/08727-0) and CNPq(162334/2021-4).

References:
[1] MANIGLIA, B. C. et al. Int. J. of Biol. Macrom., v. 138, p. 1087, 2019.
[2] TOVANI, C.B. et al. Acta Biom., v. 92, p.315,2019.

3D printing of asymetric membranes based on cationized chitosans and beta-chitin whiskers tobiomedical applications

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3D printing of asymetric membranes based on cationized chitosans and beta-chitin whiskers to biomedical applications
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Direct ink writing (DIW) 3D printing was employed to produce asymmetric membranes based on N-(2-hydroxy)-propyl-3-trimethylammonium chitosan chloride (QCat), beta-chitin whiskers and genipin as crosslinking agent with potential biomedical applications. Chitosan was produced from beta-chitin by using ultrasound-assisted deacetylation (USAD) [1] and acidolysis of beta-chitin was carried out to produce whiskers. The QCat derivative was synthed through the reaction of chitosan with glycidyltrimethylammonium chloride (GTMAC) in slightly acidic medium. The ink for the DIW 3D printing was produced through the solubilization of QCat in beta-chitin whiskers aqueous suspension, followed by the addition of genipin. The membranes were printed with two layers with different geometries. The rheological behavior of the ink used for DIW 3D printing was evaluated through shear thinning, viscoelasticity and thixotropy assays for samples with and without addition of genipin. QCat showed average degree of substitution ((DS) ¯) ~ 30%, which provided water solubility in a wide pH range and antimicrobial activity against E. Coli and S. aureus, as evidenced by MIC and MBC assays. The ink for DIW 3D printing showed pseudoplasticity, thixotropy, solid-like behavior under low oscillatory strain and liquid-like behavior under high oscillatory strain, which are ideal characteristics for DIW 3D printing. The asymmetric membranes showed contact angle of 59.4° ± 7.6° for the outer layer, swelling capacity inPBS buffer ~ 35% and total degradation after 7 days of in vitro incubation with lysozyme.The asymmetric membranes showed contact inhibition against E. coli and S. aureus in thedisk diffusion test, a notable feature considering biomedical applications.

Acknowledgements: CNPq - Process No 141800/2017-18

References

[1] Fiamingo, A.; Delezuk, J. A. M.; Trombotto, S.; David L.; Campana-Filho, S. P. Ultrason. Sonochem., v. 32, p. 79-85, 2016.

AIR AND OXYGEN PLASMA PEN TREATMENT ON THE GELATINATION OF CORN STARCH

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In this work, corn starch was modified by atmospheric pressure plasma jet (APPJ) using air and oxygen gases and the properties of starch granules and gelatinized films produced from them was evaluated. Two different routes were chosen: starch granules were modified by APPJ either using compressed air (air treatment) or by using ultra pure 99.99% pressurized oxygen (O2 treatment). Contact angle and FTIR for films produced from both starches were evaluated after two different treatment times: 30 minutes and 40 minutes of treatment. The pH of a 7.5% starch slurry in water was measured before and after treatment and it was observed that air plasma led to an increase in medium acidity while oxygen plasma led to its alkalinization. After gelatinization, air APPJ treatment led to a more hydrophobic film while O2 APPJ to more hydrophilic films. FTIR was performed in the films and no clear chemical modification was detected that could explain this behavior. XRD, DSC and viscosity measurements gave us a clue of the observed phenomena and will be discussed.are being measured in order to better understand these modifications. Therefore, APPJ is a clean technology for starch modification, changing pH showing great potential for applications both for food and non-food applications.

ALGINATE SPRAYS LOADED WITH SAPONINS FROM THE SAQTA ROOT (Colignonia parviflora subsp. Biumbellata) APPLIED FOR THE DESORPTION OF IMPREGNATED OIL IN ROCKS

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The exploitation and transportation of oil by private companies or industries, where spillage accidents occur frequently, generate environmental problems that cause destructive damage to the ecosystem of the ocean and the coast. This motivates states to generate new methods for oil removal.

This work is based on the method of enhanced oil recovery from mixtures of polymers and surfactants, being alginate and Saqta root saponins substances of interest for being eco-friendly.

The experimental phase begins with the extraction of the saponins by means of hydroalcoholic extracts from the Saqta root. This crude extract of saponins will be characterized by the instrumental techniques of FTIR, 1H-NMR and LC-MS.

Commercial alginate has been characterized by the instrumental techniques of FTIR, 1H-NMR, capillary viscometry, and TGA.

For the desorption tests, stones of similar have been collected in a coast area not contaminated by oil, in Lima-Peru. The alginate sprays have been prepared through an experiment design with three variables (%alginate, %surfactant and %plasticizer), obtaining formulations with synthetic surfactant (Tween 80) including glycerol and formulations with natural surfactant (saqta saponins) including xanthan gum. Spraying was applied to the sampled stones without oil together with a $CaCl_2$ crosslinker.

The formation of the film on the stone and its characteristics will be observed after every days. The sampled stones are submerged in crude oil for a few minutes and then allowed to drain into the environment for a day (Batch 1) and for a week (Batch 2). The sprays selected from the design of experiments were applied to each batch. The films formed with and without crude oil are removed from the stone and characterized by the instrumental techniques of FTIR, TGA and SEM. A quantitative analysis of total hydrocarbons will be carried out in the films formed to measure their crude oil removal capacity.

Carboxymethylcellulose production from sugarcane bagasse: a new approach in biorefinery concept

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The production of carboxymethylcellulose from different raw materials has been carried out frequently, but obtaining it from agro-industrial residues is still little explored in terms of aspects such as performance, cost-effectiveness and sustainability. Here, sugarcane bagasse was used as raw material for the synthesis of carboxymethylcellulose (CMCb). The natural polymer CMCb was characterized and applied in a matrix for the encapsulation of the Trichoderma harzianum strain with the objective of application as a biocontrol agent. To complement the results, an estimate was made of the production cost of CMC obtained from sugarcane bagasse considering a CMCb plant attached to an established first generation ethanol biorefinery. For this, sequential treatments were initially used for the removal of hemicellulose and lignin and isolation of cellulose for the synthesis of CMCb. The conversion of cellulose into CMCb was carried out through the application of NaOH and monochloroacetic acid (MCA). The characterizations showed the chemical modifications of the materials until obtaining a CMCb with a degree of substitution of 0.44, purity of 71.3% and crystallinity of 32%. The samples were comparatively studied by FTIR, TGA and XRD. The growth and viability of free spores were compared with encapsulated fungus and the results were positive showing that the polymeric matrix allowed the growth of the encapsulated fungus and maintained its viability after a period of 5 months of storage. The minimum value of the selling price of the product calculated for the CMCb synthesis process was 16.8 US$/kg of product, this value is above the commercial carboxymethylcellulose. However, these data are important to provide as an initial guideline for the economic performance of CMC production from an agro-industrial residue and show the importance of using technical-economic tools to support decision-making in obtaining materials from waste in future biorefineries. Fapesp Proc. 2019/05154

Carrageenan beads: exploring drug delivery potential

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Polysaccharides are increasingly being used for drug delivery, and seaweeds have become a prominent source of these compounds, including carrageenan, alginate, and agar. Carrageenan (CRG), a natural sulfated polysaccharide, has thickening and gelling properties and is used in various industries. Moreover, it exhibits several biological properties, such as antibacterial and antiviral [1]. Thus, this study aimed to develop CRG beads loaded with neomycin. For bead production, a 3% CRG solution was dripped into a 4.5% KCl + 4.5% CaCl2 anti-solvent solution and stirred for 30 min. After washing, the beads were immersed in a 20 mg/mL neomycin solution for 24h. Encapsulation efficiency was determined by measuring the absorbance of the solution using a spectrophotometer. Additionally, mass uniformity was evaluated by weighing beads loaded with neomycin and without, incrementing from 5 to 50 beads, and analyzed through linear correlation between the number of beads and the weight [2]. Results showed an encapsulation efficiency of 20%, which is average for this type of bead. Both loaded and placebo beads demonstrated high mass uniformity, with a correlation of 0.999 for loaded beads and 1 for placebo. Thus, CRG beads exhibit adequate encapsulation efficiency and high mass uniformity, making this material suitable for various drug delivery applications.

Acknowledgments:

This research was funded by Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP), grant numbers 2020/11333-4 and 2021/14698-6. Thank you, Dr. Pieter De Wever and Kennet Simoens, for the help in the lab.

References:

[1] Li F, Liu Y, Cao Y, Zhang Y, Zhe T, Guo Z, et al. Copper sulfide nanoparticle-carrageenan films for packaging application. Food Hydrocolloids. 2020;109:106094.

[2] Trygg J, Yildir E, Kolakovic R, Sandler N, Fardim P. Solid-State Properties and Controlled Release of Ranitidine Hydrochloride from Tailored Oxidised Cellulose Beads. Macromolecular Materials and Engineering. 2015;300(2):210-7.

Cellulose-based Materials: Important Player in the Circular Bioeconomy

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Cellulose-based materials can play a critical role in the circular bioeconomy thriving. Forest biomass is a critical source of cellulose. However, others could be introduced in the specialized market, for example, lignocellulosic biomass, such as sisal, whose fibers have a high cellulose content. Also, the high cellulose content gives such fibers excellent mechanical properties, and, therefore, sisal is considered one of the top plant fibers for reinforcing polymeric matrix composites. Cellulose derivatization, as esterification, has been widely studied and reported in the literature, but there is still room for further exploration. Using cellulose as a macromonomer can lead to promising avenues for future research. Specifically, microcrystalline cellulose is a compelling synthesis option due to its relatively low average molar mass. In the context of those above, there has been an effort from our side to explore the esterification of sisal cellulose, aiming, for instance, to use the products in producing electrospun mats and forming functionalized films, for example, with magnetic nanoparticles. Numerous lignocellulosic fibers have been utilized to reinforce various synthesized bio-based polymeric matrices, emphasizing the application of sisal as short fibers or blankets. Biobased polyurethanes have been synthesized using microcrystalline cellulose as a polyol through multiple processes. The syntheses have formed molded composites reinforced by plant fibers, and films (with or without additives)and using different sources of isocyanate groups. Extensive characterization of all the mentioned materials has yielded promising results. All mentioned studies on cellulose-basedmaterials are in progress, always within the perspective of contributing to advancing thecircular bioeconomy. Studies on cellulose-based materials for application in the sorption ofmetals from water are currently in their early stages.

Funding: grant 2020/13703-3, São Paulo Research Foundation

Densification of starch compound for direct dosing in thermoplastic starch extrusion process

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Thermoplastic starch (TPS) is a promising biodegradable material. With the increasing development of TPS materials at an industrial level and from a technological point of view, there is a need to focus on improving the industrial processing system to make the most of its potential. A common previous mixture of starch and a plasticizer is prepared to obtain TPS which is called ***pre-mixture*** (PM). However, focus on the fine particulates of the starchy inputs, the use of PM presents a significant challenge due to its powder form which hinders the fluidity in hoppers. That approach is still an issue that requires investigation. In this work, densified compounds (DC) of starch and glycerol were produced to be directly used in the TPS extrusion process, as a fluidized alternative. Thus, different types of DC such as tablets, agglomerates and pellets were obtained. They were produced by direct dry compaction, wet agglomeration and agglomeration with a binder (gelatinized starch paste), respectively. The DC obtained were submitted to the extrusion process to obtain TPS. The DC were evaluated by dosing in a conventional extrusion hopper and characterized in terms of particle size distribution, structural analysis by SEM and thermal analysis by TGA. The TPS films, produced using both PM and DC, were also characterized through tensile tests, DRX and thermal analysis. The qualitative evaluation of tablets DC type aimed only to guarantee its formation and stability. The shape and conformation of the agglomerates were found to be dependent on the glycerol content and moisture level. Gelatinized starch proved to be effective to allow the formation of pellets. The use of densified starch feedstock resolved feeding issues in the extruder hopper. A complete breakdown of the starch granules was achieved when DC were used after extrusion. Mechanical properties showed inferior performance when compared with TPS samples using PM inputs.

Acknowledgments:
This work was supported by CAPES.

Enhanced properties of chitosan films modified withTiO₂ nanoparticles: the surface influence

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Hybrid materials that combine polymeric matrices with inorganic nanoparticles have widely studied due to their potential applications in various fields. These materials offer a unique combination of physicochemical and biological properties, allowing for the creation of materials with different characteristics. Among the polysaccharides, chitosan is a naturaland cationic polymer derived from chitin, with an excellent biocompatibility, non-toxicity, and biodegradability properties, making it a useful material for forming films. The modification of the surface of nanoparticles plays a prominent role since this property can affect the interaction of the material with biomolecules, cells, and microorganisms. For example, conventional TiO_2 nanoparticles act as semiconductors, but when their surface is functionalized with peroxide groups (TiO_2-OPM), they begin to interact with visible radiation and produce reactive oxygen species (ROS), leading to superior antimicrobial activity. In this work chitosan films modified with TiO_2-OPM and TiO_2-red were synthesized by casting, using water as solvent. The purpose was to study the influence of surface peroxo groups on the characteristics of the material by comparing the two versions of nanoparticles. TiO_2 nanoparticles were synthesized using the oxidant peroxo method (OPM), TiO_2-red was produced by heat treating TiO_2 -OPM. The materials were submitted to optical, spectroscopic, mechanical, thermal, and structural characterizations and an optimization of loading conditions and release tests for dyes was carried out. Since the nanomaterials were distributed in the chitosan three-dimensional network, their concentration and structure influenced the thermal and mechanical properties of the films.

ACKNOWLEDGEMENTS

This work was partly funded by Fundação de Amparo à Pesquisa do Estado de São Paulo-FAPESP(2013/07296-2) and (2022/10016-0), CNPq (309711/2019-3) and CAPES(88887.641038/2021-00), (88887.643328/2021-00) and (8887.828064/2023-00)

Fatty Acid Cellulose Esters (FACEs): Structure- properties relationship elucidation according to both fatty chain length and substitution degree – Toward high value-added applications

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A lot of studies on cellulose derivatives, including ours,[1] are dedicated to the determination of mechanical and/or thermal and/or structural properties of Fatty Acid Cellulose Esters (FACEs),[2] which is a great deal if we consider the investigations on the potential applications of such bio-based materials. Substitution degree (DS) and fatty chain length are the main parameters able to directly influence properties of FACE-type materials.[3] Interested in thermoplastic and hydrophobic FACE materials, the present study sums up almost 20 years of our research work, dealing with the influence of substitution degrees and fatty chain length on structure and properties of partially- and fully-substituted FACEs. A series of fatty acid cellulose esters (FACEs) with both various degrees of substitution (from DS=1.7 to 3) and side chain length were obtained by grafting aliphatic acid chlorides (from C10 to C16) onto cellulose backbone, in a homogeneous LiCl/DMAc medium. These materials were characterized by FTIR and ^{1}H NMR spectroscopies, as well as WAXS, DSC and mechanical analyses. Whatever the alkyl chains length and the DS, all samples displayed a layered structure. The alkyl chains were able to crystallize as soon as they are long enough. As the DS decreased, the plasticizing effect of the alkyl chains was less pronounced and their ability to crystallize was improved. Regarding the mechanical behaviour and chemical resistance, similar results were observed whatever the DS is. Some high added-value applications are mentioned, as materials or additives for biopolymers.

Acknowledgments: Authors thank Artois University, especially BQR, for financial support.

References

[1]L. Duchatel-Crépy, N. Joly, P. Martin, A. Marin, J.-F. Tahon, J.-M. Lefebvre, V. Gaucher, Carbohydr. Polym. 234, 115912 (2020)

[2]C. Vaca-Garcia, G. Gozzelino, W.G. Glasser, M.E. Borredon, J. Polym. Sci. B Polym. Phys. 41, 281 (2003)

[3]J. Peydecastaing, C. Vaca-Garcia, E. Borredon, Cellulose 18, 1023 (2011)

Films based on potato starch modified by dry heating treatment; one green alternative for properties improvement

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One eco-friendly technology that is gaining attention is dry heating treatment (DHT), which has the potential to modify both the size and charge of starch molecules [1]. This research explores the use of DHT-modified potato starch, produced at varying levels (130°C for 1, 2,and 4 hours, called DHT_1h, DHT_2h, and DHT_4h), to create biodegradable films using the casting technique, along with glycerol as a plasticizer and water as a solvent. The films were analyzed for their mechanical, thermal, biodegradability, wettability, morphology, and crystallinity properties. The modified membranes exhibited a more uniform morphology compared to those produced with non-modified starch. The films based on DHT-modified potato starch displayed higher tensile strength (Native: 5 MPa and DHT_2h: 25 MPa), Young's modulus (Native: 10 MPa and DHT_2h: 1000 MPa), thermal stability (Tg of Native: 50 oC MPa and Tg of DHT_2h: 59 oC), and relative crystallinity (Native: 12% and DHT_2h: 23%), while the elongation at break was lower (Native: 20% MPa and DHT_2h: 5%) than that of the non-modified starch. Moreover, there was one reduction of biodegradability rate (~ 10%) and hydrophilicity (~ 20%) of the films with an increase in the dry heating treatment time, which can be interesting from a commercial standpoint. In summary, DHT-modified starch represents a promising alternative for the production of packaging materials.

Acknowledgments:
FAPESP, Brazil (2020/08727-0) and (CNPq, Brazil) (162334/2021-4)

References:

[1] MANIGLIA et al. Current Opinion in Food Science, v. 40, p. 72-80, 2021

FINE-TUNING CELLULOSE NANOFIBRILS FOR ENHANCED PACKING PERFORMANCE VIA ENZYME-ASSISTED DEFIBRILLATION

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Cellulose nanofibrils (CNFs) have emerged as a potential alternative to synthetic polymers in packaging applications owing to their oxygen and grease barrier performance and mechanical properties. The performance of CNF films is highly reliant on the intrinsic characteristics of fibers, including crystallinity, aspect ratio, degree of polymerization, and surface area. These characteristics may be significantly altered during the CNF isolation process, and it is important to note some properties may be enhanced others may be compromised. Therefore, understanding the changes in these characteristics during CNF isolation is crucial for tailoring CNF film properties to achieve optimal packaging performance. This study utilized endoglucanase-assisted mechanical ultra-refining to isolate CNFs, and systematically investigated the variations in their intrinsic characteristics and their impact on CNF films as functions of the degree of defibrillation, enzyme loading, and reaction time using statistical design of experiments. The isolation process was optimized to isolate CNFs with desirable characteristics for packaging and minimize tradeoffs. CNF films prepared from CNFs isolated under the optimized scenarios exhibited high thermal stability (approximately 300 °C), high tensile strength (104-113 MPa), high oil resistance (kit n°12),and low oxygen transmission rate (1.00-3.17 cc·m^{-2}.day^{-1}). Therefore, endoglucanase pretreatment allowed the isolation of CNFs with lower energy consumption, resulting in films with higher transmittance, higher barrier performance, and lower surface wettabilitythan the control samples without enzymatic pretreatment and other unmodified neat CNFfilms reported in the literature. Moreover, the mechanical and thermal performance of theCNF films was not significantly compromised.

Influence of acid treatment on the properties of beta-chitin nanoparticles

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Chitin nanoparticles, often called whiskers, are usually obtained through acidolysis. This work describes the production of beta-chitin whiskers by acidolysis with HCl, $H_2SO_4$and H3PO4 along with their structural and physicochemical properties. The starting beta-chitin had very high average degree of acetylation GA≈96% as determined by ^{13}C NMR spectroscopy, as well as high viscosity average molecular weight Mv≈413,000 g/mol. Characterization of whiskers revealed that *N*-deacetylation did not occur and that acidolysis predominantly promoted the breakdown of glycosidic bonds, leading to Mv≈10,000 g/mol. The morphology of beta-chitin whiskers was characterized by TEM and AFM. The images indicate that whiskers produced by HCl and H_2SO_4resemble needles, while those produced by with H_3PO_4exhibited a spherical shape and reduced size. Structural differences were also observed by NMR and FTIR spectroscopy, especially in the case of the H_3PO_4sample. It was found that there was a conversion of beta-chitin to alpha-chitin in this case, evidenced by the presence of characteristic resonance peaks, i.e. the splitting of signals referring to C3-C5 (≈ 75 ppm) and C6 (≈ 65 ppm) and splitting of the bands of amide I (≈ 1610 cm-1) in the FTIR spectrum and also higher indexes of short-range crystallinity (≈ 89%) calculated by ^{13}C NMR. Different behaviours were also observed in the thermal degradation profiles, in which beta-chitin whiskers produced with H_3PO_4showedhigher degradation temperatures (≈ 300 ºC), whereas the other samples had peak degradation at ≈ 250ºC. None spectroscopic evidence of the presence of sulfate and phosphate groups on the beta-chitin whisker surfaces was observed. These results indicatethat the nature of the acid used for acidolysis plays an important role on the properties of beta-chitin nanoparticles, especially leading to the conversion of beta-chitin into alpha-chitinin the case of using H_3PO_4.

Aknowledgements:
CNPq - Process no 141800/2017-18

PLA-based nonwovens modified with beta-chitin whiskers and silver nanoparticles: A new biomaterial potentially useful as wound dressing

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An innovative multifunctional material has been developed and characterized based on poly(lactic acid) (PLA) nonwoven produced by solution blow spinning and electrospinning, and modified by depositing beta-chitin whiskers on a single face and spraying silver nanoparticles (AgNPs) on it. Regardless of the spinning technique, bead-free PLA hydrophobic nonwovens were produced, while carrying out the post-modification steps on one of the membrane faces turned it hydrophilic due to the presence of numerous hydroxyl groups in the beta-chitin chains. Additionally, spraying AgNPs on such a surface resulted in antibacterial activity against S. aureus and E. coli. Moreover, the PLA-based nonwovens modified by beta-chitin whiskers and silver nanoparticles exhibited high surface area and porosity (>80%), biocompatibility towards human dermal fibroblast (HDFn, stability in aqueous medium even in the presence of lysozyme, high swelling capacity in acetate and PBS buffers and improved mechanical properties. Results show that such PLA-based materials combine several advantageous features and hold great promise as a potential multifunctional wound dressings.

Acknowledgments FAPESP grant 2021/11738-7.

Production and characterization of novel mixed-mode beads for protein adsorption and purification

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Research in protein adsorption is of great interest because it is the key step of downstream purification in pharmaceutical and food industry. Different low-cost biopolymers have been examined to obtain beads with high adsorption capacity. These bio-based materials have drawn the attention of the scientific community in this application for their outstanding biological characteristics and wide availability. Hence, a study for producing different bio-based beads for protein adsorption is justified. In this work, we present a method to produce new mixed-mode beads and examine the feasibility of their application in proteins separation and purification. The composite beads were produced by combining different polysaccharides i.e. pullulan, agar and guar gum with alginate. These novel beads were first characterized by Fourier transform infrared spectroscopy (FTIR) and then their swelling ratio was calculated and analysed. Subsequently, adsorption experiments were performed to clarify whether the materials are suitable for protein adsorption. The protein adsorption was made using bovine serum albumin (BSA) (a model protein) in solution. It was found that all the beads can perform protein adsorption in the different buffer solutions. Therefore, a selection among the different composite beads considering the best performance was made. The one containing the same composition of alginate and pullulan and the one containing alginate were selected. The adsorption of the beads selected was studied in detail and the composite beads were further characterized. The adsorption mechanism is discussed, and the characterization was conducted using scanning electron microscopy (SEM) analysis, nitrogen physisorption and the quantification of charged groups via toluidine blue assay. Finally, the selected beads were employed in a protein sample derived from a bioreactor. The results obtained allowed verifying their promising use for protein adsorption separation and purification. These results suggest a promising future of sustainable polysaccharide-based beads for applications in downstreaming and purification of proteins.

Production and regeneration of sorbent material basedon kapok fibers, chitosan and castor oil: application in the cleaning of fauna affected by oil spills.

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Oil spills cause negative effects on biodiversity, requiring remediation techniques for the affected fauna. In this context, natural sorbents are research targets because they come from renewable sources and its reusability is appreciated due to economic, environmental and operational factors [1]. Thus, this research produced composites of kapok fibers, chitosan and castor oil, tested them for the removal of mineral oil from goose feathers through light and shear contact, and evaluated the regenerative potential of compression techniques, with loads of 4 to 12 to centrifugation, with rotations from 500 to 2000 rpm for5 mi vacuum filtration for 5 min and photocatalysis, through impregnation with TiO2 subjected to UV for 2 hrs. These were made with unpressed (CNP) and previously pressed (CP) composites at 3 ton. The sorptions were measured by gravimetric method. The CNP showed an average sorption of 29.95±4.84 g/g and CP 21.50±2.63 g/g. Feathers saturatedwith oil resulted in 12.42±2.31 g/g sorption, where 84.98% and 82.84% of the sorbed oil was removed by the composite. At the end of the 4 compression cycles, the CNP maintained66%, 39%, 38% of the original sorption capacity for loads of 4 ton, 8 ton and 12 ton, while the CP maintained 68%, 39% and 42%, respectively. In centrifugation, at 500 rpm, 1000 rpm and 2000 rpm, the CNP maintained 61%, 95%, 93% of the original sorptive capacity, and the CP maintained 68%, 86% and 84%, respectively. In vacuum filtration, the CNP retained 97% and the CP 90% of the oil, as the vacuum generated was not sufficient to remove the oil, due to its high viscosity. The photocatalytic activity process was not effective.
Acknowledgements:
The authors thank the Fundação de Amparo à Pesquisa do Estado de Alagoas (FAPEAL).
References:
[1] OLIVEIRA, L.M.T.M. et al. Ultrafast diesel oil spill removal by fibers from silk-cotton tree: Characterization and sorption potential evaluation. Journal of Cleaner Production, 263 (1),

Synthesis of bio-based polyurethanes with simultaneous film formation using nanocellulose asadditives

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Polyurethanes (PU) pose an outstanding share of the plastic market, and the research for bio-based routes of PU production is an essential field of study, playing a key role in the search for a more sustainable future. This work describes the solvent-free formation of PU films simultaneously with the synthesis using castor oil (CO) and microcrystalline cellulose (MCC) as polyols [1] and an aliphatic polyisocyanate resin based on hexamethylene diisocyanate (HDIr). The films were formed from different amounts of cellulose nanocrystals (CNC) and nanofibers (CNF) in the reaction mixture. The scanning electron micrographs indicated that the films with CNC and CNF were homogeneous and presented low surface rugosity. These features influenced the mechanical properties, in which the CNC ad CNF films showed higher elongation (~50%) and tensile strength at break (~5 MPa) compared to the neat PU film (~4% and 2 MPa, respectively). The thermogravimetric analysis showed the characteristic decomposition peaks of cellulose and PU bonds, with no great difference between all the films. The contact angles of the CNC and CNF films exhibited a moderate tendency of lower angles for those containing CNF, indicating slightly higher hydrophilicity. O_2transmission rates results indicated that the nanocrystalline cellulose particles lowered the O2 permeability three-fold, with slight differences according to the amount of additive. Further studies are being performed, investigating how to coat bacterial cellulose film onto the reaction mixture, aiming to produce bio-based PUs composite-type film with good tensile and O_2permeability properties. The properties of the films containing CNC/CNF are attractive for packaging applications, among others.

Funding: Support Program for Integrated Research Projects in Strategic Areas, Research and Innovation Provost, University of São Paulo Process No: 2021.1.10424.1.9

Reference:

[1] D. S. Porto, et.al., Cellulose, vol. 29, no. 11, pp. 6301-6322, 2022.

Synthesized MgO polysaccharide starch-based: Periclase phase formation for chromium adsorptionand its reuse as green pigments

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As a nutrient found in some foods, trivalent chromium (Cr^{3+}) is more stable than the hexavalent ($Cr^{6+)}$ form, which plays an important role in glucose metabolism, but in above 1.0 $mg.L^{-1}$ it becomes harmful to health, and when left free in the environment it can end up oxidizing to Cr^{6+} [1,2]. This work aimed to synthesize magnesium oxide (MgO) in the periclase crystalline phase by the gelatinization method in a reaction medium with starch cassava. The search for natural additives, such as polysaccharides, for materials synthesis, has recently gained importance due to their reported low environmental impact, higher chemical reactivity, high combustion power, reduced calcination temperature, and action asa complexing gelling agent. Samples of synthesized and commercial. MgO (S-MgO and C-MgO, respectively) were characterized by structural characterization (XRD), composition (EDXRF), spectroscopy (UV-Vis), and colorimetry before and after chromium adsorption. In the adsorption study, S-MgO and C-MgO were investigated as adsorbents in the removal of Cr^{3+} ions from synthetic wastewater. DRX results after the adsorption process show that the periclase phase was changed to brucite with basal distance (001) equal to 0.48 nm [3], producing a green pigment. After adsorption, the hybrid pigments were dispersed in white real estate paint and tested against leaching to prove the stability of chromium ions in the magnesium oxide structure, so that materials with contaminating ions and without release into the environment have a final destination.

Acknowledgments:

Capes, Fundação Araucária, CNPq, and FINEP. S.J . is thankful for a CNPq PDJ post-doctorate grant (152230/2022-0)

References:

[1] Costa, L. D. S., Pereira, F. R. D. S., De Farias, R. F., Pereira, F. C. Eclética Química Journal, 35, 157 (2018).

[2] Wang, W., Qiao, X., Chen, J., L, H. Materials Letters, 61, 3218-3220 (2007).

[3] Wypych, F. and Arizaga, G. G. C. Quimica Nova, *28*, 24-29 (2005).

The impact of osmotic pressure on the water holding capacity and herbicide adsorption of new magnetic hydrogels composed of polysaccharide and zeolite supported on poly(methacrylic acid)-co-polyacrylamide networks

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In this study, novel magnetic hydrogels were synthed by incorporating unfunctionalized magnetic nanoparticles (Fe_3O_4) and 3-aminopropyltriethoxysilane-functionalized magnetite nanoparticles ($Fe_3O_4@NH_2$) into PMAA-co-PAAm polymeric networks containing CMC and zeolite, aiming their potential use in water herbicide removal systems. For this purpose, the swelling degree and paraquat adsorption capacity were analyzed in different salt media. A substantial reduction in the swelling degree values was observed in both the pure sample and the sample with Fe_3O_4, with only 38.0% and 23.5% of their respective capacity to store water molecules with the highest tested salt concentration (0.20 mol.L^{-1}of NaCl). However, the sample with $Fe_3O_4@NH_2$managed to retain around 52.3% of its swelling degree. Similarly, in the case of paraquat removal capacity, a decrease was observed with an increase in salt concentration. In pure water, all samples showed an adsorption capacity $q$of approximately 13.0 mg. g^{-1}. However, this capacity was reduced to values below 3.0 and 0.8 mg/g for solutions with 0.20 mol.L^{-1}of NaCl and 0.15 mol.L^{-1}of $AlCl_3$. This decrease in both analyses could be attributed to the interaction between the salt ions and the hydrophilic groups of the samples, leading to a reduction in the elasticity of the polymer chains. [1] Nevertheless, despite the reduction caused by the osmotic effect, all samples exhibited the ability to remove a concentration of paraquat higher than those reported in the literature, which was above the limit accepted by the OMS. [2] Thus, the findings of this study suggest that even in unfavorable conditions, the hydrogels synthed in this work, hold promise for use in herbicide removal from water.

Acknowledgments

The authors thank to UNESP, CAPES, CNPq, FINEP, and FAPESP.

References

[1] F. N. Tanaka, et. al. Journal of Nanoscience Nanotechnology 10, 7286 (2018).
[2] Hue, N. T. et. al. Journal of Chemistry, 1 (2018).

Acetylation of kapok fibers (Ceiba Pentandra) for oilspill remediation

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Oil exploration brings environmental impacts, which end up causing oil spills which generate irreversible consequences in the affected ecosystems [1]. One of the techniques used is natural sorbents, which are mostly made up of vegetable fibers, such as sugarcane bagasse, leafy residues, sisal, coconut, sawdust and kapok fiber [2]. Therefore, the focus of this work was to promote surface acetylation in kapok fibers (Ceiba pentandra) to improve their oleophilic or oil retention character. The concentration of treatment with acetic acid solutions was varied at 1, 2, 3, 4 and 5% (v/v), with the raw samples in contact for 1 h, followed by drying at 50 °C. The sorption evaluations were carried out via gravimetric test,by initial and final mass difference after 30 min of contact time in S-10 diesel oil. The samples were characterized by FTIR and optical microscopy to assess functional and structural changes. With acetylation, there was hydrolysis of the cellulose in the fibers, making them rougher and forming a random orientation of the fibrous bundles. It was also noted that low concentrations of acetic acid led to an increase the C=O presence, and affected sorption with increase of almost 30%, and under concentrations above 2% (v/v), damage to the fibers was evidenced with the presence of folds and rupture of the tubular structures responsible by oil storage, reaching, at 5% (v/v), a sorption similar to that of the raw material.

Acknowledgements:
The authors would like to thank the Fundação de Amparo à Pesquisa do Estado de Alagoas (FAPEAL) for funding.

References:
[1] NASCIMENTO, N. R. et al. Atividade petrolífera offshore e sua relação com os impactos ambientais nos ecossistemas marinhos. Meio Ambiente (Brasil), 3 (5), 2021.
[2] OLIVEIRA, L.M.T.M. et al. Ultrafast diesel oil spill removal by fibers from silk-cotton tree: Characterization and sorption potential evaluation. Journal of Cleaner Production, 263 (1), 2020.

Analysis of the influence of chitosan molar mass on quaternization process

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Chitosan is one of the most studied biopolymers in the world, especially due to its cationic character and biological properties. However, chitosan is only soluble on a limited acidic pH range. To improve chitosan solubility in a broader pH range and increase its applications, chemical modifications can be performed, as the quaternization process, where functional groups are introduced by substitution or addition in organic compounds [1]. It is known that chitosan molar mass affects the crystallinity, solubility, permeability and mobility of the molecular chains, however, there is a lack of studies in the literature that analyzed the influence of chitosan molar mass on the quaternization process and on the final product properties. In this study, chitosan quaternization with GTMAC was assessed with chitosan of three different molar masses and analyzed regarding quaternization degree, deacetylation degree and thermal stability. It was possible to observe that the degree of quaternization increases with the increase in chitosan molar mass, while the acetylation degree increases in a similar manner when compared to the non-quaternized chitosan, regardless the chitosan molar mass. Regarding the thermogravimetry, the peaks related to thermal degradation were observed on a lower temperature for the quaternized chitosan, and increasing chitosan molar mass decreased the temperature of thermal degradation. In conclusion, it was possible to observe that the molar mass is a key factor on chitosan quaternization and should be taken into account on studies and new technologies applied to chitosan materials.

Acknowledgment: This work was supported by CAPES.

References: [1]. Pathak, K.; Misra, S. K.; Sehgal, A.; Singh, S.; Bungau, S.; Najda, A.; Gruszecki, R.; Behl, T. Polymers (Basel) 2021, 13, 1-31.

Antibacterial potential of polysaccharide-clay nanocomposite functionalized with quercetin dye

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Polysaccharides may have antioxidant, anti-inflammatory and immunoregulatory properties making them suitable when used in biological systems [1]. Biocomposites based on clays and biopolymers are materials of increasing biological interest as they show improved mechanical and physicochemical properties [2]. The use of clays as a support can increase the stability of dyes mainly due to the adsorption and intercalation capacity of organic molecules [3]. Therefore, this work aimed to develop a new stable hybrid from the incorporation of quercetin in cashew gum-modified Laponite. The solids obtained were characterized by XRD, Infrared and SEM analysis. The antimicrobial activity for possible biological application was evaluated. The results indicated high intercalation of quercetin in the modified inorganic matrix and good antibacterial activity against Staphylococcus aureus and Escherichia coli strains. This initial research shows the potential for using the nanocomposite in safety-sensitive applications.

References

[1] G.A. de Azevedo, M.C. Heinrichs, Â.M. Moraes, Cashew tree gum for biomaterials engineering: A versatile raw material in consolidation, J. Appl. Polym. Sci. 139 (2022) 1-14. https://doi.org/10.1002/app.52484.[2] E. Ruiz-Hitzky, M. Darder, P. Aranda, An Introduction to Bio-nanohybrid Materials, 2008. https://doi.org/10.1002/9783527621446.ch1.[3] P. Trigueiro, F.A.R. Pereira, D. Guillermin, B. Rigaud, S. Balme, J.-M. Janot, I.M.G. dos Santos, M.G. Fonseca, P. Walter, M. Jaber, When anthraquinone dyes meet pillared montmorillonite: Stability or fading upon exposure to light?, Dye. Pigment. 159 (2018) 384-394. https://doi.org/10.1016/j.dyepig.2018.06.046.

A Simple Method for Enhancing Chitosan Films withKraft Lignin Carbon Dots

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The increasing popularity of biopolymer-based packaging is attributed to their eco-friendliness, non-toxicity, and biodegradability, providing a promising alternative to synthetic materials[1]. Chitosan, a biopolymer with excellent biodegradability, environmental benefits, antimicrobial properties, and film-forming ability, has been widely studied for its potential as a packaging material [1]. However, its weak mechanical and barrier properties restrict its application[1]. In this research, we aimed to address this limitation by incorporating kraft lignin carbon dots into chitosan films via a simple method without plasticizers. Our approach involved adding 20 mL of 1% acetic acid to 0.2 g of chitosan and stirring it in an ultrasound tip until dissolution, followed by adding 100 uL of lignin kraft carbon dots doped with ethylenediamine. The mixture was then transferred to a polystyrene petri dish and dried in an oven at 65ºC for 24h. We conducted stress tests, FTIR, and thermal analysis to evaluate the resulting films. The films were easy to demold and exhibited a blue-green color when exposed to a light beam in the ultraviolet region (360nm), suggesting a possible photoluminescence effect from the carbon dots. The tensile strength result of 37 MPa was promising, and FTIR analysis indicated hydrogen bonding between the single OH bonds of chitosan and carbon dots. The shift in the CH peak to 1648 cm −1 indicated increased elongation C=C. Thermal analysis revealed changes in the thermal profiles of the samples with carbon dots. Therefore, our study demonstrates the potential of developing films with desirable properties using chitosan and carbon dots.

Acknowledgments:

FAPEAM, IFC/SFS, PIPE/UFPR, LCNano/UFPR, Embrapa Florestas.

References

[1] Villalba-Rodríguez, Angel M., et al. Chitosan-Based Carbon Dots with Applied Aspects:New Frontiers of International Interest in a Material of Marine Origin. Marine Drugs20.12(2022)

Assessing the Sorbent Capability of Polysaccharide-Based Materials for Oil Sorption in Polluted Environments in Alagoas State

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In 2019 there was an oil spill in Brazil that reached the coastline of 4,334 km in 11 states in the Northeast and Southeast. From this perspective, biosorbents have been highlighted as a method for removing organic contaminants, since low-cost, abundant, and biodegradable biomasses are used [1]. Therefore, the present work evaluated the sorptive capacity of natural materials taken as agricultural residues from the Brazilian northeast, such as, kapok fiber, coconut palm straw, natural and washed sugar cane straw and bagasse, corn cob, emu shank and cassava husks, simulating their uses for remediation of spills of Lubrax 20W50 motor oil, S-10 diesel and API 29 petroleum. The sorption tests were performed using an evaluation of the relative difference in mass between the initial sorbent sample and after contact with the oil, varying this time from 1, 5, 10, 15 to 30 min. Analogously, an oil capture stability test was carried out, where the sample was directed to a Shaker incubator to be shaken under rotation of 150 rpm during times of 5 and 10 min, calculating the sorption by the relative difference again. Optical Microscopy was performed to evaluate the characteristics of the structures of the materials. It was observed that emu shank, sugar cane bagasse, coconut palm straw, corn cob and kapok fibers needed to remain in contact with oil for 30 min so that there was a greater passage of oil between the structures of the samples. On the contrary, cassava peel and sugarcane straw needed only 1 minute to reach equilibrium. In addition, sugarcane bagasse, coconut palm straw and kapok should be given greater attention to oil release and possible contamination, since they had more significant oil losses between the times of 5 and 10 minutes, being 70, 17 and 15%, respectively.

References:

[1] Oliveira, L.M.T.M. et al. Ultrafast diesel oil spill removal by fibers from silk-cotton tree: Characterization and sorption potential evaluation. Journal of Cleaner Production, 2020.

Assessment of HPAC Solution Bleaching Efficiency in Purifying Pineapple (*Ananas comosus L.*) Leaves

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Pineapple (*Ananas comosus L.*) leaves are a significant waste product generated after fruit harvesting, resulting in a considerable amount of biomass available for use. The fibrous material obtained from pineapple leaves exhibits a higher cellulose content than wood fibers and a lower lignin content than other commonly used alternative fibers such as palm and coconut[1]. Therefore, this agro-residue has great potential as a reliable and sustainable source of cellulose for producing a wide range of derivative products. However, a bleaching process is necessary to improve the quality of the obtained pulp. In this study, pineapple leaves were milled and subjected to a bleaching process using a hydrogen peroxide and acetic acid (HPAC) solution at a 1:1 ratio. The process was conducted in an oven at 80°C for12 hours. The obtained pulp was then neutralized and dried. To evaluate the efficiency ofthis process, infrared spectroscopy (FT-IR) and X-ray diffraction (XRD) were performed. TheFT-IR analysis indicated a reduction of absorbance in lignin (1595 cm^{-1}) and hemicellulose(1730, 1625 cm^{-1}) characteristic bands[2] after the HPAC treatment. The crystallinity indexeswere determined using the method described by Sang Youn O *et al.* (2005). The "in nature"PLP had a crystallinity index of 15%, while the bleached PLP had a crystallinity index of17%. Based on the results, it can be concluded that the HPAC treatment effectively reducesthe hemicellulose and lignin contents present in the pineapple leaf powder. However, the treatment does not appear to have a positive effect on removing amorphous regions, indicating the need for additional treatment.

Acknowledgments:

Research Group Agroecology and Technology for Family Farming

References:

[1] Zawawi D et al. Applied Mechanics and Materials, 773-774, 1215-1219 (2015).

[2] Alfredo R et al. Industrial Crops and Products, 43, 529-537 (2013).

[3] Sang Youn O et al. Carbohydrate Research, 340, 2376-2391 (2005).

BACTERIAL ACTIVITY OF CASSAVA HYDROGEL ASSOCIATED WITH NEROLIDOL

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The use and development of biopolymers as mechanisms for releasing bioactive compounds have shown to be quite promising due to their compatibility with the environment and high bioavailability [1]. Among them, cassava gum stands out, a polysaccharide obtained from the tubers of ***Manihot esculenta***, which contains 80 to 86% starch in its composition and presents excellent hydrogel formation capacity [2]. This work aimed to synthesize hydrogels from cassava gum associated with nerolidol and study their antibacterial activity. The direct contact method characterized the material by FTIR and antibacterial activity. The characterizations confirm the formation of the hydrogel and the presence of nerolidol in its matrix. The antibacterial activity against ***S. aureus***and ***E. coli***was satisfactory with hydrogels containing concentrations from 2% of nerolidol, showing an 80% effect on their activity. The formation of hydrogels proves promising for the release of bioactive compounds.

Acknowledgments: CAPES, CNPQ, FAPEPI, LIMAV e LEB.

Keywords: Hydrogel, Release, and Polysaccharide.

References:

[1] de Oliveira, L. H., Trigueiro, P., Souza, J. S. N., de Carvalho, M. S., Osajima, J. A., da Silva-Filho, E. C., Fonseca, M. G. (2022). Montmorillonite with essential oils as antimicrobial agents, packaging, repellents, and insecticides: An overview. Colloids and Surfaces B: Biointerfaces, 209, 112186.

[2] Cui, C., Jia, Y., Sun, Q., Yu, M., Ji, N., Dai, L., Sun, Q. (2022). Recent advances in the preparation, characterization, and food application of starch-based hydrogels. Carbohydrate Polymers, 119624.

Banana Peel Powder (Musa sapientum) to EnergySource: Treatment and Characterization

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Lignocellulosic biomass (LCB) is an energy source of great impact in today''s world and can be used as a raw material in the production of biofuels and value-added compounds [1]. Thus, the objective was to verify the technical feasibility and potential of using biomass energy from banana peel residues based on the physical-chemical characterization as a possible alternative in the production of biofuel and briquette. Prior to characterization, a pre-treatment process was performed to obtain banana peel powder. The powder was then analyzed for ash content, higher calorific value (according to ASTM D5865-13), FRX, and TG. Ash content was determined to be an average of 13.95±0.065% per gram of powder. The study also measured the calorific value, which represents the energy produced as heat during the energy conversion process. The calorific value was found to be 16960±244 KJ/Kg. As for the FRX analysis, the main elements detected in the residue (powder) were K_2O (56.56%), CaO (12.52%) and SiO_2 (7.19%). Regarding the thermogravimetric profile, a significant mass loss event was observed starting in the temperature range of 148 °C and mass of 97.23 %, reaching the end of the event at about 523 °C and mass of 37.43 %, realizing a loss of 59.80%. This can be attributed to volatilization and degradation processes of the main components of lignocellulosic biomass (cellulose, hemicellulose and lignin). Based on the excellent results obtained, it is concluded that waste from banana peel is a possible alternative in the production of biofuel and briquette.

Acknowledgments:

The authors are grateful to PNPD/FAPESQ, beneficiary program Renewable Energy, for the financial support.

References:

[1] Nair, L. G.; Agrawal, K.; Verma, P. Biotechnology and Genetic Engineering Reviews, p. 288-338, 2022.

Biodegradable films based on Lobeira starch (Solanum lycocarpum): physical and barrier properties

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Lobeira starch (S. lycocarpum) is a polysaccharide widely distributed in Brazil, with relevant attributes for the production of thin films with compatibility and biodegradability. This study aimed to develop biodegradable films of Lobeira starch and evaluate the water vapor barrier properties, moisture content, water solubility, and UV/Visible light transmission. The biodegradable films were developed according to the casting technique, with starch gelatinization in water (4%) for 30 minutes at 80 ºC, glycerol was added as a plasticizer (0.25 g g-1 of starch) [1,2]. The starch extracted from Lobeira fruits presented a visual aspect similar to commercial starch, with obtaining films of continuous structure, flexible and without the presence of cracks, a behavior associated with the plasticizing effect of glycerol on the polymeric matrix. Moisture content (22.05 ± 0,12%), water solubility (29.94 ± 0,2.45%), water vapor permeability (0.42 ± 0.001 (g.mm.KPa-1.h-1.m-2), and light barrier were similar to those described for films obtained from starch from different sources, such as cassava. Thus, according to the results obtained, the starch extracted fromLobeira fruits can be a potential biopolymer for the development of biodegradable films oredible coatings for food, in addition to acting as a carrier of natural bioactive compounds forthe development of active packaging.

Cellulose/PVA/chondroitin sulfate membranes: Study of thermal stability and release of paracetamol

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Blends of cellulose/polyvinyl alcohol/chondroitin sulfate (cel/PVA/SC) have been highlighted for expand the applications of cellulose in the form of membranes, improving their physical characteristics, such as thermal stability and drug release [1]. In this work were produced ternary blends of cel/PVA/SC cross-linked with epichlorohydrin with different compositions of SC and incorporated with paracetamol. The membranes were produced by the casting technique from mixtures of aqueous solutions (3% w/v) of PVA and SC and alkaline (NaOH/urea) of cel. The membranes were characterized by FT-IR, TGA and DSC while the paracetamol release kinetics was evaluated using a Franz cell. FT-IR results showed shift of OH bands indicating cross-linking of cel/PVA/SC membranes. TGA/DTG and DSC analysis showed that increasing the amount of SC caused a decrease in the degradation temperature and indicating a reduction in the thermal stability of the membranes. The release kinetics were evaluated by Korsmeyer Peppas, Peppas Sahlin, Weibull, Lindner and pseudo-First Order models. The results showed that the kinetics data fit better to the empirical Weibull model. The parameter b of the Weibull equation indicates a mechanism of paracetamol release controlled by Fickian diffusion (0.35 smaller b smaller 0.75)[2]. Acknowledgments: This work was carried out with the support of the Coordination for the Improvement of Higher Education Personnel - Brazil (CAPES) and Araucária Foundation/Brazil. CMLP - ESPEC and Prebiotic chemistry Laboratory (LQP). [1] MASCARENHAS, A. R. P.; SCATOLINO, M. V.; et al. Industrial Crops and Products, Elsevier, v. 188, p. 5353, 2022. [2] MIOTKE, M.; STRANKOWSKA, J.; et al. Polymer Bulletin, Springer, v. 77, p. 483-499, 2020.

Characterization of ginger bagasse – Composition, morphology and preliminary study of bleaching

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Abstract:
One of the by-products generated after ginger starch (GS) extraction consists of a fibrous material containing cellulosic fibers and residual starch. This mateiral is denominated ginger bagasse (GB). This study aimed to characterize the ginger bagasse (GB) concerning chemical composition, structural and morphological aspects, and initial studies about bleaching. Ginger processing waste has been used as a bioabsorbent for the treatment of water used in textile industries that uses malachite and methyl violet dyes [1,2]. In this work, we focus on the characterization of this material for the future obtainment of cellulosic nanostructures and their applications as reinforced polymers in the context of sustainable materials developing from biomass attributing added value to this kind of agro residue. The initial results indicated success in obtaining cellulosic material with a lower teor of residual starch and fibers rich in cellulose (TAPPI T13m-54, TAPPI T19m-54, and TAPPI T19m-54 standards) - 48.41 ± 2.93%. The original morphology (investigated by Scanning Electron Microscopy) showed that the presence of vegetal cells reinforced the necessity of the bleaching process.

Acknowledgments:
The authors would like to thank SISNANO/MCTI, FINEP, Embrapa AgroNano research network and the National Nanotechnology Laboratory for Agribusiness (LNNA) at Embrapa Instrumentation and the Federal University of Technology - Paraná and Federal Universityof Mato Grosso (UFMT- CUA) for the general facilities.

Reference:
[1] Ahmad, R.; Kumar, R.; Adsorption studies of hazardous malachite green onto treated ginger waste. Journal of Environmental Management, 91, 1032-1039, 2010.
[2] Kumar, R.; Ahmad, R. Biosorption of hazardous crystal violet dye from aqueous solution onto treated ginger waste. Desalination, 265, 112-118, 2011.

CHARACTERIZATION OF RED PROPLIS MICROSPHERES WITH SODIUM ALGINATE BYSCANNING ELECTRON MICROSCOPY (SEM)

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Propolis is a complex mixture of resinous substances collected by bees (Apis mellifera) from different plants, which allows its consistency, texture, and color to vary [1]. Scanning Electron Microscopy (SEM) allows for reaching magnifications greater than 10,000 times Optical Microscopy [2]. It is the most suitable technique for microphotographs of samples, due to the depth that the electrons can scan [2]. The objective of this work was to characterize microspheres containing red propolis with sodium alginate by SEM. The study took place in the microscopy laboratory at the IFAL Campus Maceió. A Vega 3 TESCAN apparatus was used, operating at 15 kV, with increments of x100, x200, x500, x1000, x2000, and x5000, to obtain photomicrographs of the tested formulations, with a working distance of 15.97 mm. The results of the 8 treatments demonstrated, in general, that a good part of the microspheres presented spherical shapes and, with some fissures, grooves, and/or depressions in their external walls. In formulation H, better morphology was verified, being more defined and compatible with the shape of microspheres for the use of the material as a nanocarrier of active substances.

Thanks: The Federal University of Alagoas - UFAL and the Federal Institute of Alagoas - IFAL.

References: : [1] PINTO, L. M. A. Do PRADO, N. R. T. De CARVALHO, L. B. Propriedades, Usos E Aplicações Da Própolis. Revista Eletrônica de Farmácia, v. 8, n. 3, p.76 - 100, 2011.

[2] LIRA, F. R. de. Síntese de um eletrólito polimérico sólido em forma de membrana autossustentável - alginato de sódio e perclorato de lítio. Dissertação (Mestre em Química), Pós-Graduação em Química da Universidade Federal do Rio Grande do Norte, Natal, p.95,2020.

Chitosan substrate developed with slot-die coating technique as a platform for ammonia gas sensors

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Currently, the use of harmful materials to the environment in the manufacture of devices is considered one of the significant challenges when developing electronic devices, thus the search for new substrates for flexible electronics, in general, has intensified. The material used here as the substrate is based on chitosan, a biopolymer from the polysaccharide family, obtained through the deacetylation of chitin (chitin is the second most abundant natural biopolymer on earth, second only to cellulose). In this work, we developed new flexible substrates based on chitosan and studied depositions of organic semiconductor materials dispersed in aqueous media, such as PEDOT:PSS. Here, the slot-die coating technique was used to deposit reproducible thin chitosan films, producing thin films with controlled thicknesses of ≅ 10 µm. The relationship of stability of chitosan substrates through wettability was analyzed, revealing a hydrophilic characteristic in relation to the time of 5s. The optical properties studied are through scanning electron microscopy (SEM) of thin films in which the morphological properties are reviewed, exposing its smooth andcontinuous surface. The mechanical and electrical properties are studied through the bending method, to obtain the resistance in relation to the flexibility of the conjugatedpolymers on top of the chitosan substrate. As an application for this work, biodegradablegas sensors for ammonia detection are developed. This work can contribute to the futuredevelopment of sensors for the detection of ammonia gases, thus expanding the applicabilityof the composites used.

Creating 3D Highly Porous Interconnected Bacterial Cellulose-based Architectures Through Bioconfinement Engineering for Health Sciences

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Nanocellulose membranes derived from bacteria, known as bacterial cellulose (BC), have attracted great attention in biomedical applications due to their outstanding properties, such as biocompatibility, high purity and porosity, good water holding capacity, and high mechanical strength. In addition, the intertwined chains of this polysaccharide resemble the structure and dimensions of collagen networks, making it an excellent candidate for wound dressings and tissue engineering. While the traditional synthesis method can produce mechanically robust hydrogel fibrous structures based on cellulose, we have proposed an innovative platform with remarkable properties and features. We employed bioconfinement engineering to construct highly porous, interconnected three-dimensional (3D) architectures. Specifically, superhydrophobic particles (*i.e.*, polytetrafluoroethylene – PTFE), are utilized to restrict the growth of nanocellulose-producing bacteria (*Komagataeibacter medellinensis*) in a culture medium, forming the called liquid marbles. After 5 days, PTFE particles are removed, producing highly porous BC capsules. This method enables the synthesis of biomaterials with high water content and precise control over thickness, morphology, shape, and compartmentalization. The variables that influence the conformation and in-situ growth of 3D attractive objects (*e.g.*, incubation time, the of the particles used for bioconfinement, volume of culture medium, ambient temperature and humidity during the process) are discussed in detail with respect to the air-water interface. Promising results are presented for several advanced implications in health sciences, particularly in tissue repair and regenerative medicine. In conclusion, our bioconfinement engineering method has the potential to pave the way for the development of novel biomedical devices through easy and scalable production cycle.

Development of a controlled-release device fortreatment of periodontal disease based on thermoplastic starch and chlorhexidine

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Periodontal disease is an infectious-inflammatory disease that affects the supporting tissues of the teeth. It is characterized by loss of attachment of the periodontal ligament and destruction of adjacent bone tissue, which may lead to tooth mobility and, in more advanced stages, to tooth loss [1]. Treatment requires the removal of bacterial plaque through non-surgical mechanical treatment of crown and root scaling and planning [2]. Adjuvant therapy to mechanical treatment, using antimicrobials, has been suggested in order to prevent the recurrence of the infection [3]. The aim of this research is develop a controlled-release device to be used as an adjunct to the treatment of chronic periodontal disease. In this work, Thermoplastic cornstarch (TPS) films with chlorhexidine were prepared by casting in different percentages (0.5, 1, and 2% m/m) nanoparticles of clorexidine (CHX). The films were covered with a layer of hexamethyldisiloxane (HMDSO) plasma to evaluate their physical-chemical performance and compare these results with films without HMDSO plasma. The morphological characterizations were carried out by SEM and AFM, showing good adhesion of the nanoparticles to the thermoplastic matrix, corroborating the FTIR results. Tensile properties were statistically evaluated using the Kruskal-Wallis and Dunn tests. The incorporation of nanoparticles of CHX did not lead to significant changes in strength, maximum stress and strain at break. The highest modulus of elasticity values was found for the TPS. The films TPS/CHX showed good mechanical chemical, and morphological performance, proving to be promising to be used as an adjunct to the treatment of chronic periodontal disease. Acknowledgements:
Faperj

References:
[1] AAP - American Academy of Periodontology, Journal of Periodontology, v. 86 (7), 838 (2015).
[2] J. Lindhe, T. Karring, M. Araújo. Guanabara, Koogan Ltda (2010)
[3] Teixeira, K. I.R. Dissertation of UFMG, (2008).

Development of antimicrobial bio-based film from the combination of bacterial cellulose and silver nanoparticles obtained by plant extract of Astrocaryum aculeatum (Tucumã)

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Due to its unique characteristics, such as high crystallinity index, porosity and biocompatibility, bacterial cellulose (CB) has been explored in several areas1. Silver nanoparticles (AgNPs) are known for their bactericidal properties and currently alternative methodologies have been gaining prominence, such as the use of plant extracts as reducing and stabilizing agents2. The present work aims to produce a biofilm from the combination of AgNPs obtained through the plant extract of tucumã (Astrocaryum aculeatum) with bacterial cellulose prepared from commercial fermented teas (kombucha). For the production of CB, green tea was used as a growth medium and later refined sugar was added with the addition of the propagation matrix (Scoby). For the preparation of AgNPs, 5 mL of plant extract and 50 mL of $AgNO3$ (0.1 mol L-1) were mixed with the reaction medium at pH 9.0. Then the BC (2x2 cm2) was added in the dispersion of AgNPs in aqueous medium. The UV-VIS technique was used to observe the alteration of the intensity of the plasmon band of AgNPs. UV-VIS analyses showed a gradual decrease of the plasmon band from the first to the seventh day of reaction (0.68-0.38) and also a blue shift in the maximum intensity of the silver plasmon band. Additionally, SEM results confirm the incorporation of these AgNPs in the BC surface. The results suggest that AgNPs are adhering to the surface of the biofilm, however, there is possibly a greater incorporation of larger particles into the membrane. Antimicrobial activity was detected against E. coli and S. aureus.

Acknowledgments: MCTI, FINEP, FAPESP, CNPq and CAPES.

[1] MORENA, A. G. et al. Cellulose, v. 26, n. 16, p. 8655-8668, 2019.

[2] DOS SANTOS, S. S. et al. JOURNAL OF THE BRAZILIAN CHEMICAL SOCIETY, v. 35, n. 5, p. 705-712, 2022.

Development of Bioactive Textile Coatings forEnhanced Viral Protection in Masks

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The emergence of the COVID-19 pandemic has highlighted the need for effective personal protective equipment, particularly face masks, to limit the spread of the virus. However, contamination of masks through interpersonal contact or handling poses a challenge that requires the development of bioactive textile coatings that reduce the need for mask changes and increase user protection. In this study, we report the development of polypropylene masks coated with organic antimicrobial agents, including polyethyleneimine (PEI), lauric acid (Lau), besides Cu2+ salts, and their virucidal performance against coronaviruses. The materials were characterized by Water Contact Angle and X-ray photoelectron spectroscopy (XPS). Coating virucidal performance was tested against coronaviruses (MHV-3 strain) and the cytotoxicity to L929 control cells as previously reported [1]. Water contact angle and XPS results validated the masks surface modification. The virucidal results showed that the PEI and Lau coatings led to significant viral titer reductions of 99% to 99.99% after 2 h of contact with the virus. Furthermore, the PEI coating associated with copper salt exhibited rapid action, with a viral inhibition from 99.9% to 99.99% in less than one minute, indicating that CuSO4 can improve the virucidal effect of the coating on a large scale. Although the coatings demonstrated virucidal kinetics performance compared to other antiviral coatings, the presence of copper ions potentializes their effect, promoting the virus inactivation in less than one minute. This hybrid coating proves to be a suitable candidate for applications on masks for rapid inactivation of the virus, thus contributing to the development of effective personal protective equipment in the fight against the COVID-19 pandemic.

Acknowledgements:

The authors thank CAPES and FAPESP for the financial support.

References:

[1] Rogério A. Bataglioli et al, Journal of the American Ceramic Society 105., 1748-1752 (2022).

Development of Polysaccharide-Based Hydrogels for Cationic Dye Removal

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The presence of dyes in water is one of the main sources of pollution and represents a major environmental concern, promoting the search for new adsorbent materials to treat industrial effluents [1]. In this sense, this study aimed to produce hydrogels based on polysaccharides: cellulose/alginate (CEL/AL) and carboxymethylcellulose/alginate (CMC/AL) for removing methylene blue dye (MB) in aqueous medium. The hydrogels were produced from solutions of CEL, AL, and CMC, with 50/50 (w/w) proportions of the polymers and gelled in calcium chloride solution (1% m/v). Scanning electron microscopy (SEM) analysis showed that the CEL/AL 50/50 hydrogel has an irregular spherical external morphology, while the CMC/AL 50/50 has a more compact surface. The MB dye removal capacity was up to 77.79% for the CEL/AL 50/50 hydrogel and 67.30% for the CMC/AL 50/50. The non-linear Freundlich model had a better fit for the CEL/AL 50/50 hydrogel, showing a favorable sorption with the n constant within the range of 1 to 10 and an R2 of 0.979, indicating that the adsorbent surface is heterogeneous and that has adsorption sites in different layers. for the CMC/AL 50/50 hydrogel, the Sips model provided an R2 value greater than those found in the Freundlich and Langmuir adjustments, with an n value greater than 1.0 (n = 4.117), indicating that simultaneous adsorption may occur in mono and multilayer [2]. Acknowledgments: this work was carried out with the support of the Coordination for the Improvement of Higher Education Personnel-Brazil (CAPES) and Fundação Araucária/Brazil. CMLP-UEL-LMEM/Laboratory of Microscopy and Microanalyse. References:[1] RAI, P.; MEHROTRA, S.; PRIYA, S.; GNANSOUNOU, E.; SHARMA, S.K. RAMDHAN, T.; CHING, S.H.; PRAKASH, S.; BHANDARI, B. Trends in Food Science Technology. v.106.150-159, 2020.[2] REDLICH, O.; PETERSON, D. L. Journal of Physical Chemistry. v. 63.1024-1024, 1958.

Development of simple formulations using Hydroxypropyl Methyl Cellulose and Xanthan Gum with rheology compatible with field fluids

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The preparation of fluids used in the primary cementing process of oil wells is a critical step. These fluids are responsible for promoting better stability and safety of the well. In general, the preparation of drilling fluids and spacer fluids requires complex formulations with several abrasive additives, which increases its difficulty to dispose of. Aiming to develop drilling and spacer fluids with rheology compatible with the specifications provided by PETROBRAS, two fluids with simpler formulations were developed, in order to represent the behavior of field fluids. The spacer fluid is water-based, while the drilling fluid is soybean oil-based. The preparation of the spacer fluid is done by adding the additives Xanthan Gum, HPMC, and salt and then stirring until fully dissolved. The oil-based drilling fluid was prepared similarly, using EVA (ethylene-vinyl acetate), a copolymer used in lubricants, as the main additive. The use of EVA replaces the use of bentonite, barite and microemulsions, which add complexity to the formulation and require furthermore concern in its disposal. Rheology tests were performed using a Fann Model 35A viscometer and a Mars III Rheometer, which provided data to evaluate behavior curves to indicate similarity with the fluids used in the field. The results obtained for both fluids were acknowledged as non-Newtonian behavior curves according to a Herschel-Bulkley fluid, demonstrating a close rheology with PETROBRAS field fluids according to graphics and comparative tables for tension, shear rate and viscosity. Therefore, based on the rheology results obtained, the formulated fluids were considered suitable for conducting tests that simulate primary cementing, with the advantage of their less complex, non-abrasive formulation, potential for reuse, and ease of disposal.

Acknowledgements: FUNTEF- PR, LabReo, PETROBRAS

References:

Fink, Johannes. Petroleum Engineer’ s Guide to Oil Field Chemicals and Fluids. Third Edition. Leob

DEVELOPMENT OF SUPERABSORBENT HYDROGEL USING NATURAL CHICHA GUM (*STERCULIA STRIATA*) WITH ACRYLATE

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Hydrogels classified as superabsorbents have great potential for applicability, from agriculture to environmental remediation [1]. It is due to the water retention capacity hundreds of times, thus allowing a prolonged water supply to the crop through the plant roots and beyond the control of release in the soil [2]. Thus, hydrogels were developed using highly available natural sources in the Northeast region, such as chicha gum (*Sterculia striata*) copolymerized with acrylates, for application in agriculture. The hydrogels were characterized by X-ray Diffraction (XRD), and changes were observed in the semi-crystalline profile of the hydrogel with the insertion of potassium phosphate and chicha gum. In the results through Fourier transform infrared spectroscopy (FTIR), the characteristic bands of the formed polyacrylamide, interactions with chicha gum, and the presence of phosphorus bonds coming from the fertilizer. In the results obtained by Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS), changes were observed in the material's morphology and evidence of the presence of potassium phosphate in its structure. In the swelling results, it was observed that the hydrogels have characteristics of superabsorbents, with the best swelling for the material formed from polyacrylamide and chicha gum in the proportion of 2.0/0.01 (w/w), with values of 36000.00% of swelling concerning the initial weight of the hydrogel.

Acknowledgments: The authors thank CAPES, CNPq, and FAPEPI.

References

[1] Haddad AM, Al Asadi IJ, Falih I a Q (2017) Preparation of Nano Triblock Co-polymer for Desferoxiamine Delivery and Cross-Linked Copolymer for Iron Overload Disease. Polymer science 03: https://doi.org/10.4172/2471-9935.100020

[2] Neethu TM, Dubey PK, Kaswala AR (2018) Prospects and Applications of Hydrogel Technology in Agriculture. Int J Curr Microbiol Appl Sci 7:3155-3162. https://doi.org/10.20546/ijcmas.2018.705.369

EFFECT OF THE ADDITION OF MAGNETITE NANOPARTICLES IN CELLULOSE AND ALGINATEBEADS ON THE REMOVAL OF CADMIUM IONS

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The indiscriminate disposal of toxic metal ions in water resources has been a risk to human health due to contamination of living organism through food chain. The biodegradable adsorbents are an alternative renewable, sustainable, economical, for wastewater treatment. This study investigates the effect of magnetite (MAG) incorporation in cellulose (CEL) and alginate (ALG) matrices for cadmium (Cd^{2+}) adsorption. The MAG synthesized by co-precipitation was dispersed in the proportion of 30% (w/w) in the polymer solutions 3% (w/v). CEL and CEL/MAG beads were prepared by dripping in $H_2SO_4$10%(v/v) solution and ALG; ALG/MAG; CEL/ALG and CEL/ALG/MAG by ionic gelation in $CaCl_2$3% (w/v). The beads were characterized by XRD, swelling capacity (SW) and Cd^{2+}ion removal efficiency. The additional ALG cross-linking provided by Fe^{2+}/Fe^{3+}ions from MAG is responsible for the lower SW values in beads containing ALG [1]. The highest SW values were observed for the CEL (61.85%) and CEL/MAG (70.13%) beads, attributed to the large availability of CEL and MAG hydroxyls for interaction with water. Beads with ALG have a Cd^{2+}removal range between 48.76% and 99.93%. Removal is related to the amorphous property of the adsorbents and the effect of ion exchange between $Ca^{2+}$and Cd^{2+}[2]. The highest crystallinity of the CEL and CEL/MAG beads (78.68% and 41.44% respectively) hinder the access of the adsorbate to the active sites of the adsorbent, resulting in a lower efficiency of Cd^{2+}removal (19.35% and 11, 70% respectively) [3].

Acknowledgments: This work was carried out with the support of the Coordination of Improvement of Higher Education Personnel-Brazil (CAPES), Fundação Araucária-Brazil, CNPQ and CMLP - LARX/UEL.

References:[1] UYSAL, E. et al. Materials Chemistry and Physics, v. 281, p. 125843, 2022.

[2] BOUKHALFA, N. et al. Bulletin of the Chemical Society of Japan, v. 94, no. 1, p. 122-132, 2021.

[3] YANG, J. et al. Carbohydrate polymers, v. 111, p. 768-774, 2014.

Enzymatic oxidation of xylan and lignin-carbohydrate complexes from grass-lignocellulosic materials with potential application for hydrogels and adhesives

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The occurrence of hydroxycinnamic acids (ferulates and p-coumarates) in xylan molecules extracted from grasses can be explored for laccase-mediated enzymatic cross-linking of the polymers. Lignin fractions and lignin-carbohydrate complexes contaminating xylan preparations also serve as substrates for laccase-mediated oxidations. In both cases, the increased molar mass of the polymers is expected in the reaction products, which present potential applications for the preparation of polysaccharide hydrogels and adhesives. The current work aimed to extract and transform xylan fractions from two different grass biomass materials (sugarcane bagasse and corn stover). Extracted samples were treated with laccase targeting cross-linked products. Sugarcane bagasse and corn stover, previously ground in ball mills to increase enzymatic digestibility, were treated with alkaline xylanases. Extracted xylans were recovered at 13.2% (m/m) and 14.3% (m/m) yield, respectively. Ferulate (FA) content in extracted xylans corresponded to 1.8 FA/100Xyl and 2.3 FA/Xyl, respectively. After laccase treatment, both samples presented increased molar mass in the polysaccharide and aromatic fractions. Accordingly, phenolic hydroxyl contents decreased in both samples: 0.73% to 0.16% in sugarcane xylan and 0.58% to 0.34% in corn stover xylan. Both data suggest that phenolic groups were oxidized by laccase treatment resulting in cross-linked products with increased molar mass.

Evaluation of polyelectrolyte complexes based on sodium alginate and chitosan designed for targeted colon delivery.

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Polymeric nanoparticles (NP) are being studied increasingly due to their benefits, such as nanometric, controlled release, improving the solubility and stability of hydrophilic and lipophilic active substances, vectorization, and minimizing side effects. Both chitosan (CS) and sodium alginate (SA) are used to obtain NP due to their natural, non-toxic, biocompatible, biodegradable, and mucoadhesive properties[1]. Polyelectrolyte complexation (PECs) involves interactions between polyelectrolytes with opposing charges by supramolecular interactions between CS and SA [2]. NP anionics can be beneficial sincethey adhere to positively charged proteins in inflamed tissues and can be strategic for inflammatory bowel diseases (IBDs), which are characterized by chronic inflammation in the mucosa of the gastrointestinal tract, mainly in the colon region. Mesalazine (MLZ) is an anti-inflammatory medication, but its limited solubility prevents it from reaching the colonial region [3]. This study aims to evaluate how the development of QS and SA-based PECS is impacted by pH, polymer ratio, and sequence of addition. It also assesses methods that investigate how pH affects zeta potential in polymer dispersions and MLZ as a tactical tool for colon-specific release. The results showed that the order of addition had an impact on the of NPs, while NPs with higher concentrations of polycation formed aggregates and/or s with larger PDIs, while an excess of SA decreased the and PDI of the NPs. The chart of zeta potential in relation to pH showed a useful tool for selecting the appropriate pH for the creation of PECs.

References:

[1] B.L. Banik, P. Fattahi, J.L. Brown, Wiley Interdiscip. Rev. Nanomedicine Nanobiotechnology. 8 (2016)

[2] T. Yilmaz, L. Maldonado, H. Turasan, J. Kokini, J. Food Eng. 254 (2019)

[3] A.M. dos Santos, S.G. Carvalho, A.B. Meneguin, R.M. Sábio, M.P.D. Gremião, M. Chorilli, J. Control. Release. 334 (2021)

Fucose-containing sulfated polysaccharide from *Spatoglossum schröederi* functionalized gold nanoparticles: A potent modulator of malignant phenotype of melanoma cells

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Melanoma are the most aggressive skin cancer type with highest lethality. The treatment is still a challenging issue because the systemic toxicity and drug resistance. The use of less toxic compounds can bring strategic solutions. Sulfated polysaccharides are an example of this compound, as a Fucan A extracted from a species of brown seaweed that showed antitumoral effect [1]. To refine treatments for cancer, some questions must be raised: How to target this compound to the tumor microenvironment, thus avoid systemic loss of the compound and reduce the dose for therapeutic? Looking for alternatives to answer these questions, nanoparticles use becomes interesting. In recent years, studies in nanomedicine have demonstrated the great potential of using these nanoparticles to treat different diseases, especially cancer. The objective of this work is to obtain gold nanoparticles functionalized with Fucan A and to evaluate its antitumor activity in cell culture and in animal model. The synthesis method selected to obtain the nanoparticles presents high yield, easy reproducibility and guarantees very stable dispersions for long periods. The characterization of the functionalized gold nanoparticles was performed using UV-visible light spectroscopy, transmission electron microscopy, dynamic light scattering, Fourier transform infrared spectroscopy. Nanoparticles do not show cytotoxicity for non-tumor and tumor cells and seem to be able to modulate the tumor malignancy profile by altering the invasive and migratory capacity of melanoma cells. Nanoparticles with Fucana A conjugated to a fluorophore were also obtained to evaluate the action mechanism of nanoparticles in melanoma cells. The results have shown that nanoparticles functionalized with Fucana A have great potential for therapeutic application, enhancing the antimelanoma action of this polysaccharide.

Acknowledgements:
CNPQ, CAPES, CTAF-UFPR, UFRN

References:
[1] Amorim, Int J Biol Macromol, Dec;93, 57 (2016).

INFLUENCE OF SSO SURFACTANT ON THE SOLUBILITY AND COLOR PARAMETERS OF CASSAVASTARCH FILMS

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The search for replacing petrochemical plastics by biopolymers has been increasing every year due to environmental issues. One of the disadvantages of biopolymers is that these materials are highly hydrophilic. Some additives are used to modify the hydrophilicity of films [1,2,3], which may also influence their solubility and color parameters. Based on this, this study aimed to verify the influence of the surfactant SSO (saponified sunflower oil) on the solubility and color parameters of cassava starch films. The films were prepared by solubilizing cassava starch in water, at a concentration of 2%, under agitation, at a temperature of 70°C, until the formation of gel. In addition to glycerol, used as a plasticizer,at a concentration of 20%, on a dry basis, the surfactant SSO was also added, at a concentration of 1 and 3%, on a dry basis. The filmogenic solutions were deposited in rectangular petri dishes and placed in an oven at 50°C for 5 hours. The formed films were submitted to solubility and color analyses. The results showed that the action of the surfactant on the films did not significantly influence the color parameters and solubility of the films.

Fundings: CNPq.

References

[1]. Oliveira, V.R.L.; Santos, F.K.G.;Leite, R.H.L.;Aroucha, E.M.M.; Silva, K.N.O. Use of biopolymeric coating hydrophobized with beeswax in post-harvest conservation of guavas. Food Chemistry 259 55-64, 2018.

[2]. Xavier, T.D.N.; Oliveira, V.R.L.; Leite, R.H.L.; Aroucha, E.M.M.; Santos, F.K.G. Filmes biopoliméricos baseados em fécula, quitosana e cera de carnaúba e suas propriedades. Revista Matéria, v.25, n.4. 2020.

[3]. Monteiro, A.K.S.; Oliveira, V.R.L.; Santos, F.K.G.; Barros Neto, E.L.; Leite, R.H.L.; Aroucha, E.M.M.; Silva. K.N.O. Improvement of Thermal Stability of Cassava Starch Films from the Incorparation of Bentonite Clay. MATERIALS SCIENCE FORUM (ONLINE). 2019.

LOW-COST EXTRACTION PROCESS OF ZEIN FROMCORN GLUTEN MEAL 60 AND ITS ADHESIVES BY CHEMICAL MODIFICATION

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Adhesives play a vital role in a range of industries, including packaging, construction, and healthcare. However, traditional adhesives often contain harmful chemicals and are not biodegradable, leading to environmental concerns. Zein protein offers a promising solution to these issues due to its unique properties such as biocompatibility, biodegradability, low toxicity, and good adhesive properties. In recent years, researchers have explored the potential of zein protein as a sustainable and eco-friendly alternative to traditional adhesives. Zein protein-based adhesives have shown good adhesion properties on various surfaces such as paper, cardboard, glass, metal, and wood [1,2]. In this study, we used a low-cost scalable process for raw zein extraction from corn gluten meal 60 percent, and developed zein-based adhesives by chemical modification. Zein was characterized by biuret test, TGA, DSC, and UV spectrophotometry (270 nm). Adhesives were prepared by dissolving the raw zein into sodium dodecyl sulfate aqueous solution (200 mmol/L), and treated with two different metal chloride aqueous solutions ($FeCl_3$ and $CaCl_2$). Adhesive bond strength was measured in paper card (276 g/m^2grammage) using an Instron UniversalTest equipment. Results showed good adhesive strength for both metal cations, confirmingthe potential of zein as an eco-friendly alternative to traditional adhesives.

Acknowledgements:
The authors acknowledge the São Paulo Research Foundation (FAPESP) (Process number 2022/16745-4), and Coordination of Higher Level Staff Improvement (CAPES) (finance code 001) for the financial support.

References:
[1] Y. Wei, J. Yao, Z. Shao, X. Chen. ACS Sustainable Chemistry and Engineering, v.8, p. 7668-7679 (2020).
[2] T. M. Lutz, C. Kimna, A. Casini, O. Oliver Lieleg. Materials Today Bio, v.13, 100203 (2022).

Carboxymethyl cellulose and carbon dots-based nanocomposite hydrogels for woman's health treatment

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Woman population are the higher number in Brazilian communities (over 50 %). In this context, this Glendale has several health problems caused by different factors, such as social inequalities, family' s responsibilities, and others. [1]. Nowadays, innovative materials based on hydrogels are used to treat diverse diseases. These hydrogels are a three-dimensional network with an incredible capacity to adsorb huge amount of fluids and remain these chemical structure stable [2]. In addition, in these networks it is possible to incorporate nanoparticles that can improve several properties, such as luminescence. In this sense, the carbon dots became a new "star" of Carbon Family, above all when they are produced using a green and low-cost chemical route. Considering that, this research produced new nanocomposite hydrogels based on carboxymethyl cellulose (CMC) incorporating carbon dots performed by an eco-friendly and low-cost chemical route for woman health treatment. These hydrogels were fully characterized by FTIR, UV Visible,XPS, XRD, PL spectroscopies, SEM, and TEM. Moreover, swelling and gel-fraction were evaluated. Furthermore, cytotoxicity and fluorescence based on microscopy confocal were performed. The results showed that the main chemical groups stabilized the carbon dots in CMC were hydroxyl groups. Additionally, the morphology of hydrogel presented a homogeneous spread of carbon dots, as the biological analysis showed a uterine cancer detection based on bioimaging. Then, these nanocomposites hydrogels showed a great potential for applications in Woman health treatment.

Acknowledge: FAPEMIG (APQ-02565-21), CNPq, CAPES, UFVJM

[1] BRASIL. Ministério Da Saúde, Instituto Sírio-Libanês de Ensino e Pesquisa. Protocolos da Atenção Básica: Saúde das Mulheres, 2016. Disponível em: https://bvsms.saude.gov.br/bvs/publicacoes/protocolos_atencao_basica_saude_mulheres.pdf. Acessado em: 01/03/2023.
[2] PEPPAS, N.A. LANGER, R. Science, v. 263, p. 1715- 1720, 1994.

Solanum lycocarpum starch : extraction, characterization and use in the development of films

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Polymers are sustainable, technological and economic alternatives, intended for the production of films with applications in food industry, medicine, engineering as well as other áreas. This work aimed at the extraction and characterization of native starch from Lobeira(Solanum Lycocarpum), as well as the development of biodegradable films based on this biopolymer. Films were characterized with the following techniques: scanning electron microscopy (SEM), swelling, thickness , and FTIR spectroscopy. The combination of biopolymers (PVA + starch) led to thin films of high transparency with a thickness of 55.93 ± 4.14 µm. As for the FTIR of starch, there was the presence of a broad band at 3277 cm −1 , typical of the −OH bond elongation vibration, common in native starches, as well as peaks at 1149 cm −1 , 1077 cm−1 and 997 cm −1 associated with the COC and C−OH bond elongation vibratio of polysaccharides. In the FT-IR spectra of films, an increase in the −OH band of about 3200 cm was observed, related to the interaction between the −OH groups of PVA and starch. An increment of the bands at 1149 cm −1 − , 1077 cm −1 and 997 cm −1 , related to the COC and C−OH stretching vibration of the glycosidic bonds was also observed. In terms of swelling, rapid swelling of the films was observed after 5 minutes of contact with water, after which it occurred slowly up to 10 minutes, with maximum of 59.3%. This behavior may be associated with the hydrophilic characteristics of PVA and starch. Spectra indicate the starch as type B, with a high amylose content. There is indication of good compatibility between the polymers, which opens the possibility to use and apply it as biodegradable packaging.

Monascus-Derived Pigments as a Sustainable and Eco-Friendly Colorant for Textiles and Thermoplastic Materials

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Monascus biopigments have been widely used in various industries due to their naturalorigin, stability, and color quality. In the textile industry, they are utilized as a natural dyefor fabric coloring, providing eco-friendly alternatives to synthetic dyes. Monascuspigments have also been employed in the production of thermoplastic materials, such aspolypropylene and polyethylene, to improve their color properties. This work showed thepotential of Monascus pigments produced in sugarcane bagasse hemicellulosic hydrolysatesas colorant alternative for different areas. The produced pigments derived from sugarcanebagasse hemicellulosic hydrolysates showed high thermal stability (7.213 Kcal.mol -1).Additionally, these biopigments have shown potential in the development of activepackaging materials, as they possess antioxidant and antimicrobial properties that canenhance food preservation. Overall, the application of Monascus biopigments in differentindustries is a promising and sustainable approach towards achieving a greener future.

The authors gratefully acknowledge to Coordenação de Aperfeiçoamento de Pessoal de NivelSuperior- Brasil (CAPES) Finance Code 001, Fundação de Amparo à Pesquisa do Estado deSão Paulo (FAPESP) Process number 16/10636-8, This work is part of theproject “INCT Yeasts: Biodiversity, preservation and biotechnologicalinnovation” , funded by Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Brasília, Brazil, grant #406564/2022-1

Nanocomposite hydrogels based on carboxymethyl cellulose incorporatedcellulose nanocrystal using a sub-wear out fiber from Brazilian semiarid region

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Among several natural polymers, cellulose and its derivatives have been widely used in the production of hydrogels [1]. Hydrogels are three-dimensional structures capable of absorbing large amounts of fluids. In addition, they can incorporate nanoparticles to improve important characteristics, such as mechanical behavior, thermal and electrical properties [2]. One of these very interesting nanoparticles are cellulose nanocrystals (CNs), which have interesting properties, such as biocompatibility, biodegradability, low density, high mechanical and chemical resistance, high crystallinity and high surface area, makingthem interesting for various applications [3] . The most used extraction process of CNs arechemical routes based on strong acids that cause the hydrolysis of the amorphous region ofcellulose. In this sense, this research carried out a synthesis of hydrogels based on CNsincorporated with carboxymethylcellulose (CMC) produced by two chemical routes from afiber from the Brazilian semi-arid region. These hydrogels were characterized using FTIR,UV Visible, XRD spectroscopy, SEM and TEM. In addition, swelling and gel fraction wereevaluated. The results showed that the chemical crosslinks were the hydroxyl group in citricacid and CMC. Visible UV showed bands associated with the incorporation of CNs. Furthermore, the morphology analysis demonstrated that the CNs were homogeneously spread in the hydrogels. These results showed the great potential of these hydrogels and thepossibility of intensifying the social and economic development of the Brazilian semi-aridregion.

Acknowledgment:

The authors thank FAPEMIG (APQ-02565-21, APQ-03088-2, APQ-00370-22),CAPES, CNPq and UFVJM for financial support to the project.

References:

[1] THOMAS, *et al.* Journal Mech Beh Biomed Mater, v. 110, p. 103884 (2020).
[2] BORSAGLI, *et al.* J Environ Chem Eng, v. 9, 105491 (2021).
[3] HALIB, *et al.* Materials, v. 10, p. 977 (2017).

Polyhydroxybutyrate/Babassu Biocomposites:Statistical Analysis of mechanical properties

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PHB is a biodegradable semi-crystalline polymer and originates from bacterial fermentation of sugar cane. Although PHB has similar mechanical properties to polyolefins such as polypropylene (PP), its widespread application is limited because of its high cost and certain inferior material properties such as brittleness and low impact resistance [1]. Babassu represents natural and cost-effective filler originating naturally from a palm tree found in the North, Northeast and Midwest regions of Brazil. Usually, its shell is discarded and burned without any benefit. In polymer composites babassu yields both, material and technology related improvements as well as economic benefits [2]. In this work, PHB/Babassu biocomposites were processed by compressing molding in the following wt.%:100/0, 90/10, 85/15 and 80/20. The formulations were characterized according to chemical,physical and mechanical properties. Nonlinear regression models were performed to determine the influence of the biocomposite composition on the impact resistance, hardnessand tensile results. The independent variable Babassu content (B) was statistically significant in the evaluated operating range. Tensile testing showed that biocomposite PHB/Babassu 90/10 exhibits mechanical properties similar to PHB, whereas the increase of babassu concentration for 20% wt.% should reduce mechanical properties probably due tothe poor filler-matrix adhesion. The impact resistance of PHB/Babassu was slightly higher inthe 10 wt.% biocomposite, in accordance with Hardness results.

Acknowledgements:
Faperj and CNPq.

References:
[1] R. Hoffmann, D.D.S. Morais, C.J.F. Braz, K. Haag, R.M.R. Wellen, E.L. Canedo, L.H. de Carvalho, K. Koschek, Composites Part A 124, 105472 (2019).
[2] M.B.C. Vitorino, P.B. Cipriano, R.M.R. Wellen, E.L. Canedo, L.H. de Carvalho, J Therm Anal Calorim 126, 755-69 (2016).

Polyhydroxybutyrate treated by atmospheric air plasma improving water absorption properties in Thermoplastic starch films

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In this work, PHB in powder was immersed in distilled water and modified by plasma treatment using a commercial atmospheric-air plasma-jet system [1]. The TPS/PHB formulations were composed of a proportion of 95/5 (%wt). The films obtained were TPS, TPS/PHB, TPS/PHB T40, and TPS/PHB T60. Each formulation was fed into a mono-screw extruder with three temperature zones. The PHB, PHB T40, and PHB T60, in powder, were analyzed through FTIR. All PHB characteristic bands were observed in the modified and unmodified samples. However, changes in the bands around 1720 cm-1 and 1052 cm-1, related, respectively, to C=O and C-O linkage, were observed. The intensity of the bands related to C=O increased with the treatment time. Bands associated with C-O were shifted to lower wavelengths indicating, probably, a change in PHB chains. The PHB characteristics bands were not observed in TPS/PHB T40 and TPS/PHB T60; it indicated that PHB-T´s were encapsulated by TPS. The water absorption analysis revealed that the amount of water absorbed per gram of TPS in the sample decreased with the PHB addition in treated samples. The amount of water absorbed by TPS/PHB T60 was lesser than that observed for TPS/PHB T40, indicating that the interaction between TPS and PHBT changed the TPS absorption behavior. These results corroborated FTIR analysis of TPS/PHB T, where the TPS bands initially at 3304 cm-1 (related to OH group) were shifted for 3268 cm-1 and 3289 cm-1 in the samples TPS/PHB T40 and TPS/PHB T60, respectively. The dimensional analysis of samples was performed. It was observed that PHB improved the dimensional sample''s stability. In conclusion, PHB treated by atmospheric air plasma improved the TPS hydrophilicity and the dimensional stability of composites. Acknowledgements:Faperj. References:[1] V.S. Rosa, T.S. Mendonça, R.H. Mendonça, R.A. Simão, D.C. Bastos, Composites Macromol. Symp. 406, 2200041 (2022)

POLYURETHANE-TYPE MATRICES SYNTHESIZED FROM MICROCRYSTALLINE CELLULOSE AS POLYOL

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The present study investigated the formation of hybridly reinforced composites simultaneously with the synthesis of biomass-based polyurethane (PU) matrices. Microcrystalline cellulose (MCC) was used as polyol and reinforcement. Castor oil (CO) was used as an additional polyol and also to help disperse the solids reagents. The reactions were carried out with no solvent or catalyst. The isocyanate used was diphenylmethane diisocyanate. The reinforcements were sisal mats and/or short curauá fibers (3 cm long, randomly distributed), with the presence or not of MCC as an additional reinforcement. The materials were characterized by thermogravimetric (TGA) and dynamic mechanical (DMA) analyses, flexural tests, and scanning electron microscopy (SEM). The TGA analysis indicated thermal stability of all materials up to 250°C. The DMA tan delta curves showed glass transition (Tg) below room temperature, approximately 20°C and 2°C for the - unreinforced PU and composites, respectively. The flexural tests carried out at room temperature (about 25 °C) proved elastomeric characteristics for the unreinforced polyurethane and composites without curauá fibers as reinforcement. The SEM images showed good interaction at the interface matrix-fibers and homogeneous dispersity of the MCC used as reinforcement. The materials were prepared from a highly renewable content, inside the circular bioeconomy context, with potential application in various sectors, such as automotive, aeronautical, and nautical industries.

Production and characterization of chitosan particles incorporated with Essential Oil of Oregano for active packaging

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Essential oils are substances that can be used in active packaging with great consumer acceptance. However, for its use in industrial packaging production processes, it is necessary to provide for the protection of volatile compounds against evaporation, oxidation, thermal degradation and odor reduction [1]. In this work, chitosan particles were produced by ionic gelling, using sodium trimetaphosphate and sodium tripolyphosphate crosslinkers, with different proportions of essential oil of oregano (OEO) with the objective of evaluating its potential for use in active packaging [2]. FTIR and swelling analyzes confirmed the crosslinking of chitosan and indicated that swelling is influenced by the type of crosslinker and the way in which these substances interact with chitosan. Thermogravimetric analysis demonstrated that particles with encapsulated oil have greater thermal stability than pure oil, serving as thermal protection for the active substance. By Dynamic Light Scattering it was verified that the particles produced with sodium tripolyphosphate have smaller sizes, which influenced the release of oregano essential oil. For the different particles, the oil release is described by a quasi-Fickian release mechanism. The results showed that the produced particles have sufficient thermal stability to be used in the extrusion process up to a temperature of 120 °C, protecting the active substance, in addition to masking the strong odor of the OEO, making them suitable for use in active packaging.

Acknowledgements:This work was carried out with the support of the Coordination of Improvement of Higher Education Personnel - Brazil (CAPES) and Araucária Foundation/Brazil.

References:[1] M. Yousefi; V.G. Mohammadi; M. Shadnoush; N. Khorshidia A.M. Mortazavian, Food Science and Technology International, 28, 592-602, (2022).

[2] S.F. Hosseini; M. Zandi; M. Rezaei; F. Farahmandghavi, Carbohydrate Polymers, 95, 50-56, (2013).

SUPERABSORBENT HYDROGELS OF CASHEW TREEGUM WITH LAPONITE

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The use of superabsorbent hydrogels in agricultural applications contributes to sustainable development, as they can act as soil conditioners, storing greater volumes of water and reducing the waste that occurs with continuous irrigation [1]. In this way, superabsorbent hydrogels based on polyacrylamide, laponite and cashew tree gum in different concentrations were produced to investigate the swelling capacity and the rheological properties of the hydrogels. The results show normal modes that confirmed the formation of hydrogels by FTIR and that laponite is present in the crystalline network of the nanocomposites by XRD. The presence of laponite in the hydrogels increased the strength of the material based on rheological tests. With the swelling test, it was verified that the increase in pH from 4 to 7 favored the swelling of the hydrogels, however, the swelling capacity decreases with increasing amount of laponite, as the addition of an inorganic filler in the hydrogel matrix causes an increase in crosslink density, resulting in decreased water absorption [2]. In the toxicity test, it was possible to verify low mortality of *Artemia salina* nauplii in both nanocomposite hydrogels, and that materials with higher concentration of cashew gum did not show mortality in the bioassay. The results indicate that superabsorbent nanocomposite hydrogels with higher cashew tree gum content are ideal for agricultural production.

Acknowledgment:
A UFPI, FISMAT and LIMAV.

REFERENCE

1. Głowińska, A.; Trochimczuk, AW; Jakubiak-Marcinkowska, A. New acrylate/organophosphate based hydrogels for agricultural applications. New perspective and innovative concept for the use of 2 (methacryloyloxy)ethyl phosphate as a multipurpose monomer. **EUR. Polymers. J**, 2019.

2. RODRIGUES, H. *et al.* Superabsorbent Hydrogels Based to Polyacrylamide/Cashew Tree Gum for the Controlled Release of Water and Plant Nutrients. **Molecules**, 2021.

Synthesis and characterization of chemically modified babassu coconut (Orbignya sp.) biopolymers for biomedical applications

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The babassu mesocarp, obtained from the babassu palm (Orbignya sp.) has been gaining notoriety among the Brazilian resource biodiversity due to its high concentration of starch (66 %) which confers functional properties and attractive environment for chemical modifications, being a promising material in biomedical areas [1,2,3]. The objective of this work was to obtain and characterize three oxidized derivatives from babassu mesocarp. The babassu mesocarp was isolated by maceration in an aqueous medium. For the modification of the mesocarp starch, sodium periodate was used in different starch/periodate ratios (m/v). The derivatives obtained were characterized by Fourier Transform Infrared (FTIR), Elementary analysis (EA), X-ray diffraction (XRD), thermal analysis, zeta potential, scanning electron microscopy (SEM), and solubility. In the FTIR spectra, changes were observed in the regions close to 1050 and 1120 cm−1 in the spectra of all obtained modifieds, which are related to primary alcohol groups of the oxidation process. No abrupt changes in the carbon/hydrogen ratio were observed as no new functional groups were introduced; the presence of -OH groups decreased the zeta potential. The XRD analysis evidenced different crystalline patterns among the modifieds, which may be related to the degree of oxidation, corroborating with SEM micrographs. Similar decomposition profiles were observed for all materials and heating-dependent solubility. Thus, the characterizations carried out confirmed the chemical modifications of new biomaterials obtained from babassu mesocarp with potential application in biomedical fields. Acknowledgments: Oliveira, A. C. J thanks the CNPq for the Postdoctoral fellowship 164646/2020-5. References: [1]Teixeira, P. R. S. et al. Journal of Polymer Research, v. 25, p. 1-11, 2018; [2]Oliveira A. C. J. et al. Carbohydrate polymers, Available online 23 October 2020, 117226; [3]Ribeiro, A. J. et al. Carbohydrate Polymers, 147, 188-200 2016.

Synthesis and characterization of theZnS/Nanocellulose nanocomposite

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The use of residues from the agro-industrial process has been a major focus of research in recent years due to the great potential of using residual biomass not only for energy generation, but also for the extraction of compounds that can be used for higher added value purposes, such as extractives, polyphenolic compounds, among others. [1]

Parallel to this, ZnS is a semiconductor material with a series of applications in several fields, such as photocatalysis, solar cells and optoelectronic devices, it is also worth mentioning its thermal properties, due to its thermal conductivity and specific heat capacity. [two]

In this present work, it is intended to obtain nanocellulose from the husk of Pinus elliotti, through a sequence of treatments, aiming to remove the extractives through extraction in water, the lignin through an alkaline hydrolysis, in addition to the bleaching of the pulp, viaa route with chlorides. Finally, the obtained pulp is mechanically defibrillated resulting in a nanocellulose gel. The obtained FTIR spectra confirmed that the treatments were effective in their objective of removing the unwanted phases.

ZnS was obtained through a microwave-assisted hydrothermal synthesis, where the zinc and sulfur precursors were subjected to a reaction at 150°C for 25 minutes, obtaining a white precipitate at the end of the solution, where it was possible to confirm the particle in the order of 200nm by scanning electron microscopy (SEM) and the desired phase by x-ray diffraction (XRD).

At the present moment, samples of the nanocomposites are being obtained through hydrothermal synthesis with ZnS precursors and nanocellulose in different ratios.

Synthesis of films based on chitosan and protic ionic liquids to be used as wound dressing films for oral mucosa

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The oral mucosa is important in protecting against pathogens. Various lesions can occur due to various causes, including traumatic injuries and neoplasms. These wounds can lead to systemic infections, aggravating the risks. Usually, ointments are used as a protective barrier, but they have a short duration. Films with mucoadhesive and antimicrobial properties are a great alternative to ointments because they provide a barrier against pathogens, avoid infections, and facilitate rapid and better healing of injuries by prolonging contact with the mucosa [1]. Among the materials used for making a film, chitosan has shown high potential for biocompatibility and biodegradability, but with low elasticity. Therefore, the use of plasticizers, such as ionic liquids (ILs) is necessary [2]. The films were developed by dissolving chitosan in acetic acid, in the presence of ILs: 2-hydroxydiethanolamine lactate (DLa), salicylate (DSa), and maleate (DMa). The proportions of ILs tested in relation to the mass of chitosan were 10%, 35%, and 50%. The solutions were stirred for 3 hours at room temperature. The samples were characterized by swelling degree (SD), mechanical analysis, antimicrobial properties, mucoadhesion, and cytotoxicity. The SD was inversely proportional to the amount of IL added. The mechanical analysis showed a significant change in the films when ILs were added. Antimicrobial activity was only found in films containing the IL DSa at all analyzed concentrations, and they also showed higher mucoadhesion compared to the others. Regarding cytotoxicity, the films in general did not presented cellular damage and cell growth was observed. Due to better homogeneity, elasticity, antimicrobial activity, and mucoadhesion, the chitosan-based film with DSa IL appears as a promising material for use in the treatment of oral mucosa diseases.

Acknowledgements: CAPES, PPGQ-UFRGS

References:

[1] Fernandes et al. Int. J. Polym. Sci.,2018.

[2] Chen et al. J. Phys. Chem. B., 2018.

Synthesis of silver nanoparticles mediated by aqueous bark of Citrus sinensis (orange)

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Nanoparticles, especially formed by noble metals, are used in various industrial and pharmaceutical applications. Among these metals, silver has attracted attention mainly due to its high affinity and adhesion to the cell membranes of biological systems1. The biological route of nanoparticle production has advantages over the conventional chemical route, because the usual methodologies require not only a reducing species of metal ions in solution, but also stabilizing agents of the nanoparticles created that aims to avoid their coalescence and phase separation2. The present study investigated the formation of silver nanoparticles (AgNPs) through synthesis mediated by aqueous extracts of orange peel (Citrus sinensis) under different conditions, evaluating the impact of the variation of experimental conditions (plant extract concentration, pH and temperature) in obtaining the dispersions of the nanostructures. After the production of the aqueous extract of the orange peel (20 g of pulp in 40 mL of water) three dilution conditions were investigated (1:1; 1:2 and 1:4). It was found that low concentrations (1:4) in the reaction medium were not sufficient to reduce all silver ions present during the time period investigated. In parallel, high concentrations of phytochemicals (1:1) generated a possible competition of silver cations. The conditions in alkaline medium exhibited bands related to the formation of AgNPs at shorter wavelengths, according to the UV-VIS spectroscopic technique, indicatingthe occurrence of smaller and more uniform nanostructures with respect to acidic media. The DLS results also exhibited a similar trend, indicating a lower mean of AgNPs in alkalinemedium compared to samples obtained in acidic media. For the temperature variation, withthe increase of the same there was an intensification of the signal of the surface plasmo resonance band (RPS).

Tunning the chemical and electrochemical propertiesof carbon paper-based electrodes by pyrolysis of polydopamine nanofilms

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Electrochemical paper-based analytical devices are very promising toward high-sensitivity, low-cost, affordable, miniaturized, user-friendly and decentralized analysis for applications in the health field. Regarding such properties, chemical functionalization plays a pivotal role to tune sensitivity, selectivity, and surface area of electrodes (1). However, it is challenging to control different surface properties using a single functionalization route. In this work, we attempted to tune the wettability, chemical composition, and electroactive area of carbon paper-based devices by thermally treating polydopamine at different temperatures. Polydopamine (PDA) nanofilms (2) were first deposited on pyrolyzed paper electrodes and thermally treated in the range of 300 - 1000oC in a second step. After deposition of PDA, the surface is rich in nitrogen and oxygen, hydrophilic and with a high electroactive area. As the temperature increases, the surface becomes hydrophobic and the electroactive area decreases. The surface modifications were followed by Raman, XPS, laser scanning confocal microscopy, contact angle, MEV-EDS and electrochemical experiments. In addition, different chemical compositions of nitrogen species can be tuned on the surface. As a proof-of-concept, we employed the PDA treated surfaces to anchor AuCl4- ions. After electrochemical reduction, we observed that depending on the temperature used it is possible to control the size of the nanoparticles on the surface. Our route opens a new avenue to add versatility to electrochemical interfaces.AcknowledgmentsThis work was supported by LNNano, SISNano, CNPEM, UFABC and CAPES. References:1. Noviana E, Ozer T, Carrell CS, Link JS, McMahon C, Jang I, et al. Microfluidic Paper-Based Analytical Devices: From Design to Applications. Chem Rev, vol. 121, p. 11835-85, 20212. Lee H, Dellatore SM, Miller WM, Messersmith PB. Mussel-Inspired Surface Chemistry for Multifunctional Coatings. Science, vol. 318, 426-430, 2007.

Q-Materials for Additive Manufacturing

Additive manufacturing of ceramics, opportunities and challenges with emphasis on vat polymerization

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Additive manufacturing (AM) of ceramics is a reality in the production of complex, customized, and high-performance parts. Almost all additive manufacturing techniques have been tried for ceramic materials. The selection of a technique depends on the compromise between parameters and the required properties of the product. For products with high mechanical performance, precise shape and, the vat photopolymerization (VP) process by Digital Light Processing (DLP), an indirect method, has reached high levels of acceptance worldwide. It has the following key requirements: 1) formulation of a photocurable slurry with high ceramic solid loading (~50 vol%); 2) equipment that adds uniform layers of slurry and projects on its surface an image with a light spectrum of interest, polymerizing slice by slice of the part; 3) debinding with a controlled removal of the organic fraction; 4) distortion-free sintering. This work presents characteristics of the leading ceramic additive manufacturing techniques with their main advantages and challenges, emphasizing the importance of research in the early days of this recent manufacturing area.

Fapesp process: 2021/12612-7

Additive manufacturing of PLA/SEP bionanocomposites: Influence of nanoparticle content and printing conditions on crystallinity and morphology

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Fused Filament Fabrication (FFF) process is widely used as additive manufacturing (AM) technology. One of the most important challenges faced by the FFF technique is the development of new raw materials and the definition of their processing conditions [1]. There is great interest in the development of PLA (poly(lactic acid))-based materials for 3D printing mainly due to its excellent properties such as biocompatibility, biodegradability, processability and mechanical performance [2]. Crystallinity in polymeric materials is a fundamental factor to understand their performance during application, as well as their dimensional stability and shrinkage during and after forming. The crystallization of PLA depends on the type of stereoisomer used in its synthesis, the presence of additives in its formulation, as well as the conditions employed during its conformation process [3]. In this work, PLA bionanocomposite filaments with different contents of sepiolite nanoparticles (SEP) were developed through twin-screw extrusion, with subsequent conformation of specimens by FFF at different times of exposure to the thermal environment typical of the process, evaluating the variation of the degree of crystallinity in different regions of the specimens and the morphology of the printed parts. The results indicate that, for the PLA used, the crystallinity is more dependent on factors related to the composition (presence and content of SEP) than to the printing conditions analyzed. The presence and content of SEP also had a significant influence on the printability of the materials, since the higher the content, the more reproducible the process and the better the adhesion of the deposited layers.

Acknowledgements: FAPESP (2018/16747-1) and CNPq (427639/2016-6)

References:
[1] D. Pham and R.S. Gault, Int. J. Mach. Tools Manuf. 38, 1257 (1998)
[2] G. Giammona and E.F. Craparo, Molecules 23, 4 (2018)
[3] L.C. Sanchez et al., Int. J. Adv. Manuf. Technol. 105, 2403 (2019)

Holistic approach for quality assurance and quality control for laser powder bed fusion

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The fact that additive manufacturing (AM) produces the structural material and the shape of the manufactured object together, is the very basis for the huge design freedom and at the same time the reason for a fundamental quality control challenge: Historically, quality assurance and control (QA/QC) is done separately for (i) the material composition and integrity (e.g. light microscopy, EBSD, …), (ii) surface inspection (e.g. by microscopy, deflectometry, …) and (iii) form measurement (e.g. fringe projection, laser scanner, …). Additive manufacturing produces errors in all three categories: Defects like voids and cracks in the material, rough surfaces from layerwise production and form errors e.g. due to stress. In consequence any single measurement method is insufficient for characterization. Even CT, the current gold standard, is limited as a single method because of lack of penetration of high density materials and limited resolution. So, we need a multi-method approach with a correlation of the individual results. In this presentation, we will explain how in process monitoring can be used to identify anomalies and variations during laser powder bed fusion and apply CT based solutions to link process stability and part integrity. ZEISS, as a provider of metrology and inspection equipment of a broad variety, is collaborating with industrial partners to exploit this potential.

Mechanical properties of an aluminum quasicrystal-forming alloy prepared by laser powder bed fusion

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Al-Fe-Cr-Ti quasicrystalline phase former alloys are metastable materials that form a quasicrystalline phase (QC) in an α-Al matrix. The combination of the high-strength QC phase in a ductile α-Al matrix gives rise to composites with good mechanical properties inhigh temperatures. High cooling rates are needed to precipitate the QC phase, which turnsthe processing of these alloys into a challenge. The purpose of this work was to use the Laser Powder Bed Fusion (LPBF) process, which applies cooling rates around 104 K/s, to fabricate Al95Fe2Cr2Ti1 samples using gas-atomized powders produced from recycled material. The room and high-temperature (up to 500°C) tensile properties and microstructure of the LPBFed Al-Fe-Cr-Ti alloy were investigated. The LPBF led to a microstructure with nm-d quasicrystalline precipitates in a dendritic α-Al matrix. The QC phases were present mainly in the center of the molten pool, while the heat-affected zones showed coarser but also nanometric quasicrystalline and crystalline phases. The samples exhibit high thermal stability and outstanding mechanical performance with yield strength values of ~300 MPa and ~180 MPa at 300°C and 400°C, respectively. A model is proposedto predict the yield strength of the alloy, showing good agreement with the experimental results. The crack propagation mechanism was investigated, and a considerable influence of the build direction in the ductility at room temperature was observed. The results obtained show a new opportunity to fabricate metastable quasicrystal forming alloys for high-temperature applications with freedom of design and lower cost.

Microstructure of a heat-treated 420S SS deposited by LDED on AISI D2 substrate

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Laser Directed Energy Deposition is one of the processes most used for metallic materials additive manufacturing. It also can be used for coatings deposition, in which it is possible to deposit a different material of the substrate. The understanding of the microstructure formation and its evolution during the processes chain, including post-processes steps, are of fundamental importance in the development of applications of this process. The present work presents a microstructure characterization of a 420S steel deposited by LDED on an AISI D2. In addition to the as-deposited microstructure, the effects of distinct heat treatment routes on the microstructure evolution of 420S steel were evaluated. These heat treatment cycles were tempering at 300 °C, 400 °C, and 500 °C with and without the quenching step. The microstructures were analyzed by optical microscopy, scanning electronic microscopy, and microhardness measurements. The as-deposited 420S steel microstructure is predominantly made up of austenite and martensite. Tempering heat treatments directly from the as-deposited condition did not result in significant microstructure changes. However, it was possible to observe changes in the microhardness field consistent with what was expected for the tempered martensite of 420S steel. Moreover, the tempering treatments did not reduce the microhardness heterogeneities of the as-deposited material. The quenching heat treatment significantly changed the microstructure of the deposited material, probably due to recrystallization in the austenitization step, during which there was possibly carbon transport by diffusion from the AISI D2 steel to the 420S SS. This carbon transport was probably responsible for a microhardness profile formation, avoiding a step transition between the materials. The quenching and tempering heat treatments resulted in microstructures consisting predominantly of tempered martensite, as expected for 420S steel.

Selective Laser Melting of β Ti Micrometric Powdersdown to Atomic Sputtering of Thin Films for Implant Applications

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β Ti alloys are acclaimed for biomedical applications due to their mechanical properties, good biocompatibility and corrosion resistance. Based on these characteristics, pure Ti and different Ti alloys have been applied in the biomedical industry, especially the Ti-6Al-4V alloy. However, for orthopedic applications, there is a considerable difference between elastic modulus of Ti and α+β alloys, from E = 100 to 110 GPa, and that of human bone,around E = 30 GPa. Another approach is produce a coating (~1 μm thin film) over a highermodulus alloy (stainless steel - SS) in order to reduce the surface elastic modulus of SSimplant adopting low modulus β Ti-Nb-based alloy thin films. In this context, the metastableβ alloy Ti-15Nb (% by weight) was processed by selective laser melting (SLM) and arcmelting, since this is free of cytotoxic elements and shows lower Young modulus. Characterization of SLM β Ti-15Nb alloy and Thin Films of β Ti-Nb-based alloys obtained bymagnetron sputtering were done by optical microscopy (OM), X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission (TEM) coupled to ASTAR - automaticcrystal orientation mapping (ACOM). Vickers microhardness, and elastic modulus via nanohardness and impulse excitation were evaluated. The coating of Ti40Nb20Zr40 alloy(at.%) presented lower elastic modulus of 51 GPa and hardness values, and these wererelated to the increases in both the lattice parameter and mean nanoscale grain size of thinfilm. For SLM of β Ti-15Nb alloy with a α´+ β microstructure, the Vickers microhardnessand elastic modulus measured by nanohardness and impulse excitation technique were evaluated, showing suitable modulus of around 60 GPa for biomedical applications. Besidesthat, the microhardness was found around 300 HV and density, which plays an importantrule in mechanical properties, was found above 99%. Therefore, SLM of β Ti-15Nb has beenshowing a promising material for orthopedic applications.

Additive manufacturing of microstructures with nanometric resolution

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Additive manufacturing using the Two-Photon-Polymerization (2PP) technique can produce microstructures with nanometric resolution. The 2PP process involves using a femtosecond laser with a focal point of either 780 nm or 522 nm to activate a photopolymerization agent. This technique has various applications in fields such as micro-optics, micromechanics, microfluidics, biomedical engineering (including biofabrication and medical instruments), surface texturing, filters, and metamaterials. Additionally, microstructures can be built integratively on pre-existing structures, such as fiber optic tips and optoelectronic/microfluidic circuits. 3D printers capable of this integrative resource are already being commercialized. CTI Renato Archer is acquiring an I3D/2PP printer through the EMUTISAÚDE Project (FINEP 01.18.0027.00). This printer has several specific features, including the ability to use photosensitive hydrogel-based bioinks that can incorporate living cells. Furthermore, it does not use the dip-in technique, which can compromise lens durability and facilitates the development of custom photopolymers. The printer uses an adaptive voxel size that can build regions of different resolutions in the same structure, and its configuration can be upgraded to print mesoscale structures in conjunction with micro-ones. The I3D/2PP printer will be made available to Brazilian researchers by the Coordination of Technological Park and Open Laboratories (COLAB) of CTI Renato Archer by the end of 2023. This work will detail the principles of operation, types of photopolymers, and applications of I3D/2PP, aiming to encourage researchers to submit project proposals to COLAB.Acknowledgements:FINEP and CNPq - Brazilian financial research agencies

References:[1] Greant C., Van Durme B., Van Hoorick J., Van Vlierberghe J. Multiphoton Lithography as a Promising Tool for Biomedical Applications, DOI: 10.1002/adfm.202212641, Adv. Funct. Mater. 2023

A review of the modification of aluminum powders for processing by LPBF

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The aluminum alloy processing in laser powder bed fusion (LPBF) is constantly limited due to the raw material`s intrinsic characteristics that hinder the alloy processability within this process. Although casting aluminum alloys with an Al-Si(Mg) base composition, are usually easily processable within LPBF, these alloys present a low mechanical performance. In contrast, wrought aluminum alloys are characterized by optimal mechanical properties which can be optimized by applying heat treatments. Nevertheless, its wide solidification interval increases its hot-cracking susceptibility (HCS). Likewise, aluminum powders are characterized by their high reflectivity, which requires high energy inputs to guarantee the melting of the powder bed, which difficult their application by LPBF. These limitations can, to a large extent, be mitigated by modifying the raw material from the incorporation of reinforcing particles, the adaptation of the alloy composition, and the surface modification of the powder particles. Considering the above, this review contains the different strategies used to modify and design aluminum alloys suitable to LPBF, related to: 1) inoculation and reinforcing by adding ceramics and intermetallic composites, 2) Reduction of the solidification interval, and 3) surface modification, as well as their effect on the mechanical properties and improvements in the printability of aluminum alloys by LPBF. Likewise, some of the technological contributions that have facilitated the modification and design of aluminum alloys for the LPBF shall be mentioned. The compilations made in this review shall allow visualizing the recent technological advances in the area and the discussion of key issues that require attention to continue advancing within this work-line.

Computational modeling of the mechanical behavior of additively manufactured PA12.

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.Additive Manufacturing (AM) opposite from conventional subtractive processes, consists in make parts or components from a 3D model data by joining materials layer by layer. This fabrication method has gained a lot of attention and is continuing to grow and deliver critical and complex parts and components in multiple industry sectors, according to intelligence market ASTM Wholers. This technology offers several advantages comparatively to conventionally subtractive manufacturing methods, however, the understanding of the behavior of components manufactured using AM is much lower compared to that of conventionally manufactured materials, presenting additional challenges to perform more accurate analyzes. Therefore, and particularly for polymer materials, the objective of the present work is the study the mechanical behavior of the Polyamide PA12 additively manufactured using Selective Laser Sintering (SLS), submitted to uniaxial tension, through experimental testing and numerical simulations. For this purpose, a comparison of the stress-strain behavior of a standard specimen for tensile tests and a complex geometrical specimen was carried out. In addition, computational simulations using the finite element method (FEM) were performed in COMSOL Multiphysics 5.6, using a viscoplastic Chaboche constitutive model, for the characterization of the mechanical behavior of the PA12 specimens under uniaxial tensile and the corresponding experimental validation [1-3]. The results have shown excellent agreement of the implemented FEM model with the experimental test, for both cases, the standard and the complex geometrical specimens, considering a time-dependent load environment, and the methodology could be extended to other polymer materials. Acknowledgments: The authors would like to thank CNPq for the financial support of this study. References:[1] de Souza Neto, E., Peric, D., Owen, D., 2011. Computational Methods for Plasticity: Theory and Applications. Wiley.

Corrosion Resistance of AlSi10Mg Processed by Conventional and Additive Manufacturing Techniques

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Corrosion resistance is a critical requirement for materials used in a range of applications, where exposure to harsh environments can cause degradation or failure induced by high concentrations of moisture, oxygen and other chemical elements. Thus, the development of corrosion resistant materials and suitable processing techniques, to ensure durability and reliability of components in these industries is essential. This study investigates the corrosion resistance of AlSi10Mg alloys produced by Laser Powder Bed Fusion (L-PBF) and benchmarks it against the conventionally processed casted AlSi10Mg and extruded heat-treated Al 6061 T6. Cyclic polarization tests in NaCl 3.5% naturally aerated media were used to assess the corrosion resistance of specimens, tested in duplicate and in the as-polished condition. The microstructure and corrosion mechanism were investigated by optical microscopy and the chemical composition by optical emission spark. Results show that the casted AlSi10Mg and the Al 6061 T6 specimens had very similar behavior, showing a steep increase in current density after the onset of corrosion and no signs of passivation. On the other hand, L-PBFed AlSi10Mg specimens developed a more noble corrosion potential with corrosion rates at least 20× smaller than those of the samples processed byconventional techniques. Besides, these samples were also able to passivate in aggressive media, developing much smaller corrosion rates throughout the test. The superior corrosion resistance of the specimens processed by L-PBF seems to be associated to a higher purity and to the fine-grained microstructure that accounts for the formation of a denser passive layer. Overall, this study demonstrates the promising potential of advanced manufacturing techniques for producing corrosion-resistant components, with important implications for industry.

Cracking susceptibility of AISI A2 Tool Steel manufactured through Laser Powder Bed Fusion

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Tool steels are materials used for the fabrication of tools employed in the manufacturing processes of other materials (i.e. cutting, shaping, forming) and, depending on their use, require specific mechanical properties to be enhanced. These range from resistance to softening at high temperatures, to wear resistance or strength and toughness, these last two being the most important for cold-work tool steel grades [1]. Given the strict requirements for manufacturing and heat treatment of tool steels and the higher difficulty to machine complex structures into them, Additive Manufacturing (AM) through Laser Powder Bed Fusion (L-PBF) could be an alternative to conventional manufacturing methods. However, in the case of high carbon content materials, such as high-speed or cold-work tool steels, the severe cooling rates and steep thermal gradients imposed by L-PBF cause high thermal stresses to appear, which may in turn cause these materials to experience cracking [2]. In this sense, solutions have been researched in order to prevent these defects from appearing, such as baseplate preheating to reduce thermal gradients [3], or the use of different scanning strategies, in order to better distribute stresses. In this work, cracking behavior of Tool Steel AISI A2 manufactured through L-PBF was investigated by optical microscopy, for unheated and preheated baseplate at 200°C and 500°C. Microstructure was also investigated by SEM analysis and phase composition was determined by Rietveld analysis of synchrotron XRD results, for as-built samples and tempered at two different temperatures (200°C and 500°C).

Acknowledgements:
FAPESP n. 2020/09343-1, 2022/06310-0 and 2017/27031-4; CNPq n. 103361/2022-7 and 405545/2021-5.

References:
[1]R. A. Mesquita, Tool Steels: Properties and Performance, 1st ed. CRC Press, 2016
[2]F. Galbusera et al., J. Mater. Process. Technol., v.302, 2022
[3]K. Kempen et al., J. Manuf. Sci. Eng. Trans. ASME, v. 136, n. 6, 2014

Developing Ti-based multi-layered materials using a multi-material laser powder bed fusion platform

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In structural materials, mechanical behavior improvements always focus on increasing mechanical strength, ductility, and toughness; however, these properties are mutually exclusive. Generally, the enhancements in strength occur at the expense of ductility and vice versa in the context of the strength-ductility paradox. An approach to enhance ductility (and toughness), while preserving mechanical strength, is to merge different materials to produce a composite material arrangement. Additive manufacturing (AM) technology is an effective approach for fabricating metallic composite materials with enhanced mechanical strength and ductility. Laser powder bed fusion (L-PBF), also known as selective laser melting (SLM), is an AM technique that enables the manufacture of customized objects. The concept of AM, which involves constructing objects by depositing layers of powder with the ability to vary their composition, is particularly well-suited for manufacturing composite materials or compositionally graded materials. Currently, multi-material processing represents a crucial challenge in the additive manufacturing of metallic materials. This study aims to provide new insights into the manufacturing of composite materials made up of alternating layers of different Ti-based materials (CP Ti / Ti-6Al-4V alloy and Ti-42Nb /Ti-5553 alloys) using a novel multi-material laser powder bed fusion platform. Very dense layers were obtained. The evaluation of the interface between the two materials indicated intense atomic diffusion. The Vickers hardness and elastic modulus tests revealed that the values varied according to the phase and composition. The results revealed the successful production of composite materials.

Acknowledgments: We acknowledge the São Paulo Research Foundation, FAPESP, grant # 2018/18293-8, for financial support.

Development of L-PBF contour parameters andoptimized cooling channel geometry

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Maraging 300 (M300) is an high strength steel that has shown excellent response to L-PBF (Laser Powder Bed Fusion) due to its good processability, machinability and dimensional stability [1]. L-PBF processing combined with the properties of M300 is promising for the studies of cooling channels [2]. Efficient cooling channels should provide uniform cooling and reduce cooling time [3]. This work aimed to develop L-PBF parameters for the fabrication of cooling channels by varying contour parameters and channel profiles. To investigate the optimal contour parameters, octagonal prims were produced by L-PBF with three scanning strategies (bulk without contour, bulk before contour, contour before bulk), three contour power (100, 125 and 150 W) and three contour scanning speeds (400, 450 and 500 mm/s). Ten different cooling channel profiles were fabricated, varying the channel cross-sectional area and geometry (circular, semicircle and conformal). Optical microscopy (OM) and scanning electron microscopy (SEM) were used to assess the channel dimensions and surface conditions, the images were analyzed in specialized software to obtain the roughness, density and porosity. Results showed that M300 can be processed by L-PBF with high density (up to 99.98 %) and surface roughness can be reduced (Ra ≈ 8 μm) without increasing contour porosity. The optimal contour parameter was obtained by scanning bulk before contour with laser power of 125 W and scanning speed of 500 mm/s. Channels manufactured with the optimized contour parameter showed better dimension control and fit to the digital project, than channels produced without the contour scan. Results also indicated that the channels with a conformal geometry showed good surface conditions and a cross-section area that could favor the coolant flow.

References:

[1] Kuo, CC.; Jiang, ZF. Int J Adv Manuf Technol 104 (2019).
[2] Tan, C. et al. Materials and Design 196 (2020).
[3] Reiber, T. et al. Materialwiss. Werkstofftech 52 (2021).

Effect of pores on the tribological and tribocorrosion behavior of additively manufactured lattice structures

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The ever-growing orthopedic implants market is a result of the increased life expectancy. In this regard, the demand for implants with better functionality and longer lifespan also grows, creating a constant need for research in new materials and processes for orthopedical implants. One of the main causes of implant failure is aseptic loosening, caused by a mismatch between the stiffness of the bone and the material of the implant. Metallic materials are currently the most used materials in orthopedics, but even biocompatible alloys with low Young modulus still have properties far beyond the ideal. In this regard, porous materials can be a feasible alternative as they present lower stiffness when compared to solid bulk materials. Additive manufacturing allows the production of porous materials and a precise control of the pores morphology, , and interconnectivity, which are vital features to control both mechanical properties and biological response. However, these structures are complex and their behavior as an implant material is not fully understood. The human body is a dynamic and complex environment, and an orthopedic implant material is commonly subjected to various aggressive conditions, such as corrosion, wear, fatigue, and high mechanical loadings. This work produced Ti-6Al-4V porous lattice structures and solid bulk samples using electron beam melting (PBF-EB) additive manufacturing process and compared their behavior under wear and tribocorrosion tests. All the porous and bulk solid samples presented similar behaviors under wear and tribocorrosion, with no statical differences between the specific wear rates. Abrasive and adhesive wear were the dominant mechanisms of material removal. In conclusion, the presence of pores did not increase the specific wear rates of the material, but, as a effect of the porosity, a lifespan reduction under wear and tribocorrosion conditions is expected for the porous material.

EXPERIMENTAL ANALYSIS OF PARAMETERS FOR USE OF AISI H13 IN ADDITIVE MANUFACTURING PROCESS – LASER POWDER-DIRECTED ENERGY DEPOSITION

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Additive manufacturing (AM) of metals is a disruptive technology in Industry 4.0 that enables the creation of complex 3D geometries from computer-aided design (CAD). AM has been widely adopted in various industries, including aerospace, energy, and mold repair and manufacturing. Directed energy deposition (DED) is one of the AM technologies, which uses a laser beam or an electron beam to fuse powder metal or wire onto a substrate or a manufactured part. Beads are deposited on top of each other to create layers and form the desired geometry. However, due to the characteristics process, there are defects such as porosity, and lack of fusion between the layers, which can be caused by overlapping, distance between the beads, deposition strategy and loss of the laser focal point in this research, it was carried out an experimental analysis of these variables process parameters such as laser power, mas. s-volumetric flow, and scan speed. using AISI H13, one of the most common materials used in mold repair and fabrication. The samples were characterized using confocal laser microscopy, scanning electron microscopy (SEM) and, Vickers microhardness. The results show that high mass or volumetric flow rates induce porosity and lack of fusion in the depositions. The scan speed, overlapping and laser power affect the layer's dimensions, generating problems throughout the material deposition. Furthermore, it was found that the manufacturing parameters do not influence the microhardness of the component.

Exploring the Use of HDDR-Processed Powders as Feedstock for Powder Bed Fusion Additive Manufacturing of Bonded Magnets

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Rare earth permanent magnets are crucial components in various high-tech applications, such as electric motors, wind turbines, and hard disk drives. However, these magnets are typically expensive and their production relies on rare earth metals and their alloys, which are often limited in availability [1]. To address this issue, the use of hydrogenation disproportionation desorption recombination (HDDR) has gained popularity as a method to recycle and recover rare earth metals [2]. This process can produce high-quality rare earth powders from scrap magnets or other waste sources, which can then be used as feedstock for the production of bonded magnets. Additive manufacturing (AM) is a promising technology for the production of rare earth bonded magnets, with powder bed fusion beinga popular technique due to its design flexibility and material waste reduction [3]. However,the success of powder bed fusion in producing rare earth bonded magnets depends heavilyon the quality and properties of the feedstock powders used. This work explores the use ofHDDR-processed rare earth powders as feedstock for powder bed fusion additive manufacturing to produce bonded magnets. The effect of HDDR processing parameters on the magnetic properties and microstructure of the feedstock powders is investigated. Finally, the powders are used as feedstock for powder bed fusion AM to produce bonded magnets, and the resulting magnets'' microstructure and magnetic properties are analyzed and compared to those of conventional rare earth bonded magnets. The findings of this study have significant implications for the production of rare earth bonded magnets using powder bed fusion AM. Using HDDR-processed rare earth powders as feedstock could potentially reduce dependency on costly rare earth metals and offer a more environmentally-friendly approach to producing high-performance magnets.

Gas Atomization of AA2017 for Additive Manufacturing:A Systematic Investigation into Powder Production and Processing

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Gas atomization (GA) is the main method for producing metallic powders for Laser Powder Bed Fusion (L-PBF) [1,2]. Optimization of GA is crucial to achieve specific particle properties and increase productivity [3]. This study analyzed the influence of three main process parameters (atomization pressure (P); nozzle diameter (D); and superheat temperature (T)) on obtaining AA2017 aluminum alloy powders. One experiment (D=1.5mm; T=150°C; P=20 Bar) produced metallic particles with suitable flowability and density for L-PBF. Results showed that process parameters had poor influence on d50 but significantly affected the width of size distribution curves. A mathematical analysis using the Lubanska equation resulted in a good correlation between measured and calculated d50 ($R2 = 0.97$).
(Acknowledgements: Vitor Eduardo Pinotti - FAPESP n° 2017/27031-4 and FAPESP n° 2020/08145-1; Angelo Fernandes Andreoli - FAPESP n° 2021/12056-7; Piter Gargarella - FAPESP n° 2017/27031-4; Federal University of Sao Carlos, Graduate Program in Materials Science and Engineering. This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.)

Laser heat treatment of AISI M4 multilayers depositedby L-DED

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Tool repair performed by Laser Directed Energy Deposition (L-DED) has shown promising results for the tooling industry [1]. L-DED provides the flexibility to repair diverse geometries, considering the multiple tool failure modes and locations. Given that the materials used in the L-DED repair operation must have equal or better performance than the matrix, post-processing heat treatments are frequently required. Localized heat treatments, such as laser heat treatment (LHT), can be performed to avoid the costs of heat-treating the repaired tool in furnaces [2]. In this study, AISI M4 multilayers were deposited by L-DED on AISI D2 plates and the effect of LHT with different parameters was assessed. The LHT was performed with a laser spot of 3 mm x 20 mm, scan speed of 10, 15 and 20 mm/s, and temperature of 1050, 1150 and 1250 ºC. The multilayers deposited and heat-treated were analyzed by optical microscopy, scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS) and microhardness. The cross-section micrographs reveal multilayers with low porosity and no cracks. The mean hardness of the AISI M4 deposited multilayer in the as-deposited condition (675 ± 41.85 HV) and after LHT (770 ± 39.29 HV) was higher than the AISI D2 substrate (595 ± 28.75 HV). Moreover, the results show that LHT can be used in combination with L-DED in the recovery of complex tooling, providing flexibility in repair operations and improving material performance.

References:

[1] G.Y. Baek et al., Metals vol. 9, p. 282 (2019).
[2] C. Park et al., Journal of Mechanical Science and Technology vol. 33, p. 829 (2019).

Magnetic texture development approaches on Rare-Earth based composite magnets obtained via Additive Manufacturing – Prospects and Challenges

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Rare earth-transition metal (RE-TM) based permanent magnets are an important class of functional materials essential to renewable energy and e-mobility applications. Among all other ferromagnetic compounds, the Nd-Fe-B is the most important magnetic system, vital for such applications. Beyond its magnetic properties, permanent magnets based on the Nd-Fe-B can be obtained either as a polymer-matrix composite or in a bulk sintered form [1]. The possibility to combine either magnetic and geometrical features have made Additive Manufacturing (AM) techniques gain momentum and rise as potential route to fabricate functional magnetic components with tailored properties. One key step of permanent magnets manufacturing is the crystallographic texture development, where the magnetic flux is enhanced in one particular direction. Concerning AM of Nd-Fe-B composite magnets, the state-of-art reports two different approaches to realize such step, both using a magnetic torque. The first one is carried out during the AM procedure (in-situ), where the magnetization of the powder bed or a composite filament is realized. The anisotropic particles then are oriented parallel to the external magnetic field inducing magnetic anisotropy. The second method is performed as a post-printing procedure, where the as-printed magnet is heated at elevated temperatures under an external magnetic field, allowing the particles to align towards a particular direction. The complex integration of a magnetic field source into the AM process, limited crystallographic texture and severe geometrical distortions exhibited by the fabricated components are some of the bottlenecks of the reported methods. This work addresses the challenges towards the magnetic texture development routes of RE-based composite magnets obtained via AM. An alternative in-situ technique based on mechanical torque is also discussed.

References

[1] Coey, J.M.D., Engineering, 6, 119-131 (2020)
[2] Huber et al., Sci. Rep., 7, 9419 (2017)

Main parameters and challenges for producing high-quality alumina-based ceramic parts via digital light processing technique

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Additive manufacturing techniques based on vat polymerization have been receiving special attention due to their ability to produce complex ceramic parts without the high cost of molds usually required for traditional processing methods [1]. However, the design of optimized photosensitive suspensions as well as the control of the printing parameters can have a major influence on the quality of the resulting ceramic parts [2]. In this context, this work evaluated intrinsic (exposure time and layer thickness) and extrinsic parameters (curing depth, dispersant content, and heat treatments) required for the digital light processing of alumina-based ceramic pieces. Light-curable suspensions with 50 vol.% of reactive alumina and different dispersant contents were formulated and had their rheological and stability behavior analyzed. The suspension with the most promising performance was selected and various ceramic parts were printed in a commercial printer, considering distinct exposure time and layer thickness values. The permanent linear change, bulk and relative densities, mechanical strength, and microstructural features of the samples were evaluated after debinding and sintering steps up to 1550°C. A processingmap (correlating relative density, exposure time, and layer thickness parameters) was designed and analyzed. In general, exposure times greater than 4s led to ceramic parts withhigh relative density (>78%) and suitable performance, whereas shorter time.

Acknowledgments:

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brazil (CAPES) - Finance Code 001, and CNPq, Conselho Nacional de Desenvolvimento Científico e Tecnológico - Brazil.

References:

[1] Chaudhary, R. et al. Progress in Additive Manufacturing, 8, 331-351 (2023)

[2] Camargo, I. L. et al. Ceramics International, 47, 11906-11921 (2021)

Mechanical behavior of additively manufactured maraging steel samples

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This study aimed to assess how aging affects the mechanical properties of maraging steel (M300_ processed by additive manufacturing, laser-powder bed fusion technique, and aged in different conditions. Aging heat treatment was carried out at temperatures of 470 °C, 510 °C, and 530 °C, with times ranging from 0.33 to 96 h, followed by air cooling. Despite the layer-by-layer consolidation mechanism of the material, we found a weak preferred orientation in the as-built condition, and X-ray diffraction analysis revealed the presence of only martensite and austenite. After aging, our results showed a sharp increase in Vickers hardness, compressive strength, and tensile strength, while the work-hardening ability of the samples decreased. Despite using thin single-edge bending samples measuring 5 mm, the plane-strain fracture toughness (KIC) requirement was met, and we obtained valid KIC results ranging from 49 to 64 MPa.m$^{1/2}$[1]. These findings have important implications for designing new applications of high-strength steel in additive manufacturing, including thin-walled, lattice structures, and reduced cross-section parts aimed at minimizing weight across different industries.

Acknowledgments: FAPESP, CNPq, and Capes.

[1] Pedro L.L. Santos, Julian A. Avila, Eduardo B. da Fonseca, André H.G. Gabriel, André L. Jardini, Éder S.N. Lopes, Additive Manufacturing, Volume 49, 2022.

Mechanical Properties of 410L Stainless SteelProcessed by Laser Additive Manufacturing

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Additive manufacturing (AM) processes are used for rapid prototyping and production of customized parts with complex geometries. Laser Powder Bed Fusion (L-PBF) provides superior finishing and geometric precision, while laser Directed Energy Deposition (L-DED) is capable of producing parts with large dimensions at a fast deposition rate. AISI 410 martensitic stainless steel has high mechanical strenght and corrosion resistance, making it ideal for applications ranging from cutlery to pump and valve parts. To improve the processability by AM, a low-carbon content variation of this alloy (410L) is preferable. In this study, the microstructure and mechanical properties of 410L samples produced by L-PBF and L-DED were evaluated in as-built and heat-treated conditions, and compared to those of a commercial 410 substrate. The results show that all AM samples had superior yield and ultimate tensile strengths due to the fast cooling rates, resulting in a refined microstructure compared to the commercial 410 material. However, the commercial alloy had the highest elongation, indicating superior impact energy absorption. While samples produced by AM had lower impact energy absorption in the as-built condition due to directional solidification and grain boundary segregation of brittle phases, heat treatment partially recrystallized and redistributed the phases, significantly improving the material''s toughness. Heat-treated L-PBF samples had the highest toughness values of up to 70 J (compared to 40 J for the substrate), while heat-treated L-DED samples had the highest yield and ultimate tensile strengths of 674 and 845 MPa, respectively (compared to 368 and 644 MPa for the substrate). The results indicate that additively manufactured samples havea competitive mechanical performance in comparison to commercial materials, contributingto a better understanding of the intricacies of the processing and metallurgy of this material.

Non-equilibrium solidification of the ferromagnetic Fe-Si6.5 alloy: microstructure and physical properties

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Fe-Si6.5 (%wt.) is a ferromagnetic alloy with attractive magnetic properties, such as near-zero magnetostriction, near-zero magnetocrystalline anisotropy, and high electrical resistivity. These properties, in combination with the low price, make the Fe-Si6.5 (%wt.) alloy an interesting option for energy storage and transmission. However, processing difficulties related to limitations of obtaining sheets with thicknesses of 0.1-0.3 mm, which is ideal to reduce eddy current losses, made unfeasible the application of the alloy on large scale. This limitation occurs by the brittle character of intermetallics B2 and D03 phases that are formed during cooling in quasi-thermodynamic equilibrium. Recently, the processing of Fe-Si6.5 (%wt.) by selective laser melting (SLM) has been proposed, in which extreme cooling rates are achieved. In this way, the investigation of non-equilibrium solidification is of great interest. Thus, the present study evaluated the effect of liquid undercooling on microstructural evolution, phase transformations, and how these characteristics influence the magnetic properties of Fe-Si6.5 (%wt.) alloy. The metastable solidification regime was achieved using electromagnetic levitation (EML) and glass fluxing techniques. The microstructure of the solidified samples was studied by microscopy techniques and X-ray diffraction, and the thermal behavior of the samples was evaluated. The effects of rapid solidification on the magnetic properties, namely the hysteresis loops, were investigated. In conclusion, this work aims to provide a fundamental understanding of the behavior of the Fe-Si6.5 (%wt.) ferromagnetic alloy when processed by non-equilibrium solidification methods.

Parametric modeling for Additive Manufacturing: software comparison and applications

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Parametric modeling enables fast creation of models by algorithm-driven digital model generation, the basis of generative design (GD). For prototyping it allows professionals to quickly adapt models to fit their purposes, thus accelerating product conceptualization and substantiation. Although Additive Manufacturing (AM) makes it possible for complex geometries to be fabricated, a fundamental part of AM in the GD context is obtaining the parametric digital 3D models. The aim of the present work is to generate parametric structures using different softwares available and compare the process and results. The Renato Archer Information Technology Center is a Brazilian research center located in Campinas, state of São Paulo, it supports academic and industrial research with AM techniques such as SLS, FFF, EBM, Polyjet, Binderjet and Waxjet through the 3D printing Open Lab (LAprint). A methodology for generating parametric models was developed for the free softwares Blender and OpenSCAD and it was compared to the process with another softwares available, such as Grasshopper, nTopology and Altair GD solutions. Blender visual programming Geometry Nodes is similar to Grasshopper, a graphical algorithm editor that can be integrated McNeel Rhinoceros. Blender also allows the user to automate tasks through python scripting and have the feature Vertex Groups to select portions of the model to be optimized similar to nTopology features. The parametric structure generation process comparisons between softwares presented in this study summarizes the possibilities in different softwares and brings a prominent free software option for GD in the AM context. An essential feature of the digital 3D model is to be promptly adjusted to satisfy the requirements of dynamic projects and applications, achieving these goals using low cost software solutions can greatly improve research and industry applications for AM.

Processability of AISI 410L Stainless Steel by Laser Powder Bed Fusion

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Metal additive manufacturing (AM) has been increasingly used in various sectors of industry. Among the different metal AM technologies, Laser Powder Bed Fusion (L-PBF) stands out for its ability to fabricate complex geometries with high dimensional accuracy and good surface finish. 410 martensitic stainless steel is widely used in a range of applications, from cutlery to pump and valve parts, owing to its high strength and corrosion resistance. However, the material''s brittleness poses a challenge in the L-PBF process, anda low-carbon variant, 410L, is preferred to improve processability. In this work, the properties of the gas-atomized powder were analyzed, a design of experiment was performed to establish an optimized set of L-PBF processing parameters and tensile specimens were then produced to assess the mechanical properties of this alloy processed by L-PBF. The powder presented relatively spherical particles, with an average circularity of 0.679, low internal porosity, few satellite particles, the chemical composition was in accordance with the specification and the particle distribution was 27.8-56.6 μm (D10-D90).To obtain the set of processing parameters, a central composite experiment was used, varying power, scanning speed and hatching. The data from the experiment was fed into a mathematical regression and the resulting set of parameters produced a sample with relative density of 99.95%. The specimens manufactured with this set of parameters exhibited a tensile and yield strength of 681 MPa and 621 MPa, respectively, with an elongation of 13.65%. However, the as-built material was found to be brittle upon impact, with an average absorbed energy of 3.9 J, which is attributed to the microstructure with elongated grains and brittle phases segregated in the grain boundaries. However, a heat treatment route was capable of increasing the impact strength to values as high as 70 J, with a small reduction in the tensile strength, to 619 MPa.

Production of conductive filaments based on recycled PLA for the development of 3D-printed electrochemical sensors

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Additive manufacturing (3D printing) is an interesting tool for the development of electrochemical devices by using polymeric filaments with low cost, low waste, versatility, reproducibility for large-scale production, and portability [1, 2]. In addition, the combination of waste recycling and additive manufacturing allows the application of the circular economy approach. Based on this, this work proposes the use of recycled plastic waste toobtain new 3D-printed electrochemical cells and sensors. For this, waste coffee pods composed of biopolymer polylactic acid (PLA) were processed and re-extruded to manufacture conducting and non-conducting filaments. The first one was obtained by mixing PLA pellets, plasticizers, and carbon-based materials (i.e., carbon black and carbonnanotubes). Electrochemical cells and conductive electrodes were 3D-printed in different shapes and sizes, in which the cell was able to be recycled three times without printing issues. The conductive filament presented flexible and printable characteristics. The sensorprinted by using the conductive filament had better results compared to a PLA and carbonblack commercial filament, exhibiting faster electron transfer and higher electrochemicalsurface area [3]. Furthermore, the proposed sensors showed better electrochemical performance for the sensing of caffeic acid in coffee and tea samples and biosensing of yellow fever virus cDNA in blood serum samples. Thus, the produced materials showed lowproduction cost, thermal stability, and high-quality printability. Finally, this work reports thesynergism between sustainability, additive manufacturing, and electrochemical detections,aiming to achieve the circular economy concept.

Acknowledgments:

FAPESP, Capes, and TRANSFORM-CE.

References:

[1] R.M. Cardoso, C. Kalinke, et al. Anal. Chim. Acta 1118, 73-91 (2020).

[2] M.J. Whittingham, et al. Talanta 4, 100051 (2021).

[3] E. Sigley, C. Kalinke, et al. ACS Sust. Chem. Eng. 11, 2978-2988 (2023)

Residual stresses investigation in Metal Additive Manufacturing

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Additive manufacturing supports advances in materials'' processing adaptability using a disruptive way to fabricate complex-shaped structures by adding material systematically. Given this, the Laser Powder Bed Fusion (LPBF) is a technology capable of producing near net shape components based on fusion bondings obtained from a laser-based fusion of metallic powder particles. Even with the evolution of technologies involved in the process, controlling some critical attributes of the material processed with LPBF, like residual stresses, remains a matter to deal with during process planning. Residual stresses are intrinsic to material processing, but their components'' magnitude and behavior - tensile or compression - depend on the manufacturing route and its conditions. Regarding LPBF, thermal stresses are commonly expected due to the technology operation source. Thus, it is important to consider this property for LPBF parts to avoid residual stresses undesirable to the component''s final application. This study characterized the surface residual stress of maraging steel, an alloy applied in structural and tooling industries, using the of X-ray diffraction (XRD) technique. Differences in surface residual stresses could be seen depending on the processing conditions, and the main mechanisms identified for the residual stress generation were discussed. Finally, the proposed analysis contributes to LPBF process planning.

Retained austenite decomposition pathways in additively manufactured H13 hot-work tool steel

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In laser beam powder bed fusion (PBF-LB) of AISI H13 tool steel, the as-built microstructure develops a microsegregated solidification structure of elongated cells, in which retained austenite (RA) decorates the cell walls and the interior contains martensite. The inhomogeneous distribution of alloying elements locally stabilizes RA and affects the phase transformations upon heat treatment. Synchrotron X-ray diffraction, scanning electron microscopy, and atom probe tomography were used to study the RA decomposition kinetics in the 550-650 °C range. The as-built condition contains circa 20-26 vol% of RA due to microsegregation of C, Cr, V, and Mo. During heat treatment, two microstructural pathways were observed. At low temperatures (T 650 °C), isothermal transformation of RA into ferriteand coarse carbides occurs within 600 s of the isothermal soaking. Martensite overtempering occurs, along with the dissolution of the solidification structure, promoting softening.

This work was supported by FAPESP, CNPq, and CAPES. This research used resources of the LNLS/CNPEM (XTMS beamline) and LNNano/CNPEM (SEM and TEM facilities). APT experiments were performed at the KNMF/KIT. Specimens were produced at Omnitek.

Self-disinfecting photopolymerizable nanocompositesfor 3D printing: allying antiviral properties

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Nanostructured materials and their mechanisms of antiviral activity present efficacy in coatings and surface treatments for an expressive group of viruses [1]. Nanocomposites with self-disinfecting properties act in an active-passive way for applications in materials whose objective is the inhibition of microorganism growth and viral inactivation [2,3]. Thiswork reports the development of a self-disinfecting material based on copper nanoparticles(CuNP), copper oxide (CuONP), and hybrid phase (CuNP + CuONP) incorporated in photopolymerizable resin. The production of these photopolymerizable nanocomposites for 3D printing with potential self-disinfecting properties for antiviral activity aims to inhibition of virus propagation. Allying bioinspiration, these materials allow the exploration of different hierarchical structures by 3D printing technology through active-passive mechanisms. Therefore, CuNP and CuONP were synthed by two methodologies (chemical reduction via L-ascorbic acid and mechanochemistry) and presented average s of 18.3 nm (CuNP) and 8.3 nm (CuONP). The photopolymerizable nanocomposites were characterized by UV-vis, FTIR, DRX, and SEM. Antiviral tests were performed in collaboration with FIOCRUZ-PE, initially with a model virus (Zika virus), in a NB-2 biosafety level environment, and later tests with SARS-CoV-2 will be performed. Preliminary results show that the nanocomposites produced have antiviral activity and were able to inactivate the model virus analyzed.

Acknowledgments:
This work was supported by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) RIMA Project - Edital Capes 09/2020 and CNPq Brazilian Agencies.

References:
[1] A. Bhatti, R. K. Delong, ACS Pharmacol. Transl. Sci. 6, 220-228 (2023)
[2] E. Toledo et al., PLoS ONE 17, 1-12 (2022)
[3] A. P. Mouritz et al., Nano Select 2, 2061-2071 (2021)

Smart additives based on nanoparticles of mixed oxides derived from LDH in the crystallization of phases formed during cement hydration

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Synthetic clays known as lamellar double hydroxides (LDH) are a class of multifunctional materials that, when added to cement, can contribute to the formation of the microstructure of hydrated cement. The thermal decomposition of LDH (400 500 °C) leads to the formationof nanocrystalline mixed oxides (MO), which can regenerate the lamellar structure in contact with water and anionic solutions, through the memory effect. The regeneration of the LDH lamellar structure directly influences the rheological behavior of the cement paste after the addition of MO particles, with the acceleration of paste consolidation in the first hours, indicating the potential application of these materials for 3D printing. Investigations using the time-resolved X-ray absorption spectroscopy demonstrated that the recovery of the ZnGa-HDL nanostructure was driven by a mechanism of nucleation and aggregativegrowth. In simulated concrete pore solution (pH 14) the formation of the LDH phase wasverified. Reconciling the memory effect and surface area (37 m2g□1), MO particles can actas nucleation points, contributing to the strengthening of the hydrated cement microstructure. For this, the formation of the hydration phases was monitored by X-ray diffraction for the cement paste containing ZnGa-LDH and the respective MO during 4 h andthe results demonstrated the acceleration of the hydration process, evidenced by the increase in the formation of the ettringite (AFt), especially for samples containing MO, indicating that regeneration of the LDH structure favors the kinetics for obtaining AFt. These results are interesting, since the AFt, formed in the first hours, is converted over timeinto hydrated calcium monosulfoaluminate (AFm), which has a layered structure similar tothat observed in hydrotalcite-type compounds. This makes it possible to correlate the effectof MO addition particles in the hydration phases with the structural changes of the cement.

The role of rheology in the development of bioinks for3D bioprinting

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The development of bioinks for 3D bioprinting relies heavily on rheology, which involves the study of flow and deformation of matter. Bioinks, specialized materials that act as ink in 3D printers, are used to precisely position living cells in complex patterns and shapes to create functional tissues and organs. To ensure successful 3D bioprinting, the rheological properties of bioinks must be carefully optimized. This involves ensuring that the ink flows smoothly through the printer nozzle [1], remains stable after deposition, and has mechanical properties that are compatible with the surrounding tissue environment [2]. Here, we evaluated the rheological properties, such as viscosity, storage modulus G’ and loss modulus G’’ of alginate-derived bioinks with different degrees of bioinorganic ion-mediated crosslinking, which are crucial for successful printing. By assessing the bioink over a wide range of process parameters, including temperature, pressure, feed rate, and nozzle geometry, we were able to determine its printing behavior. Our findings revealed that the network strength, shear-thinning, yield point, and thixotropy are key factors that govern bioprintability. Our research provides a comprehensive approach to bioink development anda highly reproducible and well-characterized procedure for printing with the newly developed hydrogel. Additionally, our work provides detailed insights into the relationship between printing parameters, rheological parameters, and short-term cultivation stability.
Acknowledgments: The authors are grateful for the financial support provided by the São Paulo Research Foundation (FAPESP), through processes 2022/07967-3, 2022/11767-0, and 2022/03247-6. This research was carried out at the Laboratory of Biomaterials and Biointerfaces (BBLab) of the Aeronautics Institute of Technology.
References: [1] A. Schwab et al. Chem. Rev. 120, 11028-11055 (2020);[2] M. E. Cooke and D. H. Rosenzweig. APL Bioeng. 5, 011502 (2021).

The Thermo-Calc Additive Manufacturing Module: An integrated Calphad-based FEM tool

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Today's state-of-the-art Finite Element (FEM) tools tend to use relatively simplified materials descriptions using handbook values that might have little dependence on chemistry or even temperature. Thermo-Calc has spent the last few years developing new models to predict thermophysical material properties to incorporate with CALPHAD-based materials descriptions. This is currently used to extract CALPHAD-based materials data for use in dedicated FEM codes and is now laying the foundation for our development of a completely integrated tool to simulate Additive Manufacturing (AM) processes. The AM Calculator is released in Thermo-Calc version 2023b, and it gives a unique possibility to address the problem of solidification during AM, where we obtain a unified treatment of both process parameters and chemistry-dependent thermophysical properties when solving the multi-physics problem of a moving heat source that melts and solidifies metal powder. The updated Scheil calculator allows for pre-generating a solidification profile, now also including evaporation properties, that can be saved into your own materials library, or used directly as input to the AM Calculator. Examples of the AM module applied to different material classes are shown.

3D-printed covalent immobilization-based carbon black immunosensors for portable electrochemical detection of SARS-CoV-2 spike S1 protein

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3D printing technology has gained ground due to its wide range of applicability [1]. Recently, 3D printing has played a significant role in the fight against the global pandemic event COVID-19 through decentralized and on-demand manufacturing of different personal protective equipment and medical devices, such as masks, valves, and swabs [2]. However, 3D printing technology is still considered recent and with great potential to be explored for the most diverse purposes, including the manufacturing of analytical devices [3]. The development of new conductive filaments contributes significantly to the production of improved electrochemical devices. In this sense, the fabrication of 3D-printed devices for the development of electrochemical (bio)sensors has become an almost unexplored field of great interest. Therefore, this work has presented the production of a new 3D-printed electrochemical immunosensor, based on the covalent immobilization of antibodies on electrodes obtained from lab-made conductive filaments, composed of carbon black and polylactic acid, for the detection of the spike S1 protein of the SARS-CoV-2 virus. It was possible to create an immunosensor without the use of metallic particles since the surface of the additively built sensor had readily available carboxylic groups. Under the optimized parameters, the immunosensor presented a linear range of 0.01 to 4.5 nmol L^{-1}, and a detection limit of 2.7 pmol $L^{-1}$for spike S1 protein. Therefore, a highly viable option to be used as a point-of-care device, the 3D-printed immunosensor was entirely developed for in loco application and all findings were obtained from portable equipment.

[1] Cardoso, R.M., et al. Anal. Chim. Acta, 2020, **1118**, p. 73-91
[2] Choong, Y.Y.C. et al. Nat. Rev. Mater., 2020, **5**, p. 637-639
[3] Muñoz J., Pumera M. Chem. Eng. J., 2021, **425**, 131433

3D printing of activated-carbon-zeolite porous composites from sugarcane bagasse ash for CO_2 adsorption and H_2 purification

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To overcome the environmental concerns related to climatic changes and limit the atmospheric concentration of carbon dioxide (CO_2), improved gas-separation processes such as pressure-swing-adsorption (PSA) through the use of adsorbents for CO_2removal and H_2-purification are of great interest. In this research, sugarcane-bagasse ash (SBA), an agroindustrial by-product, was used to manufacture a sustainable adsorbent from biomass-activate-carbon/zeolite porous composites using 3D-printing techniques. This alternative type of adsorbent was prepared by incorporating from 1.0 to 10.0 wt % of SBA-activated carbon into the SBA-zeolite-porous monoliths, followed by hydrothermal treatment in order to improve the specific surface area and micro-meso porosity characteristic, as well as improve CO_2affinity. In addition, SBA-derived zeolite-porous monoliths were 3D-printed with designed configuration to be used as standard-adsorbent for comparison. Optimized SBA-activate-carbon/zeolite-inks containing different additives (2.5-10.0 wt.%) of halloysite clay (HCT), as plasticizer, (1.0-4.5 wt.%) of methylcellulose (MC), as thickener and (1.0-5.0 wt.%) of poly(ethylene glycol) (PEG), as lubricant, respectively, were prepared. The three-dimensional monoliths were freeze-dried at (-100 to -15°C) for 1 h and dried for 24h at ambient conditions. Afterward, samples were fired at 650 °C for 1 h at a heating rate of 10 °C/min. Fired monoliths were transferred to a Teflon capsule containing an alkaline solution (2.5 wt.% of $Al(OH)3$ and NaOH 3M) and surfactants species (sodium dodecylbenzenesulfonate) in order to driven 13X-zeolite structure, and then, deposited into stainless-steel autoclave at 105°C ± 5°C (for 72-160 h) to provide the precipitation of zeolite crystals in situ. The specific surface area ~564 $m^2g^{-1}$was determined by Brunner- Emmet-Teller analysis (BET). Preliminary results indicate the viability of these SBA- composites as CO_2capture and H_2-purification.

3D printing of hydrogels for treatment of cutaneous leishmaniasis

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Leishmaniasis is a tropical disease caused by the parasite Leishmania and transmitted through sandfly bites. The current treatment options are limited due to the high toxicity of most drugs. However, 3D printing (3DP) technology has emerged as a promising approach for treating leishmaniasis. Tea tree oil (TTO), derived from the *Melaleuca alternifolia* plant used for its medicinal properties, has antimicrobial properties against various bacteria, viruses, fungi and parasites [1]. The purpose of this work was, from 3DP, to produce nanocomposite hydrogel *scaffolds* composed of alginate, cellulose nanocrystals, and collagen with incorporated TTO for the treatment of cutaneous leishmaniasis. To our knowledge, the use of 3DP to obtain a hydrogel construct for treating cutaneous leishmaniasis has yet to be reported in the literature. In vitro studies were carried out to investigate the activity of TTO dissolved in culture medium after 24 hours of incubation. Half maximum effective concentration (EC50) was obtained with 0.08% TTO against L. amazonensis and L. braziliensis promastigotes. Against RAW 264.7 macrophages, half maximal cytotoxic concentration (CC50) was 0.14%, indicating approximately 2-fold greater selectivity against the parasite. In the evaluation of the impregnated scafolds, a dose-dependent response was observed with the inhibition of 90%, 85% and 70% of L. amazonensis promastigotes after 24h of incubation with scafolds impregnated with 10%, 5% and 2.5%, respectively, of TTO. Tea tree oil has shown good leishmanicidal activity and is a promising treatment against cutaneous leishmaniasis.

Acknowledgments: Conselho Nacional de Desenvolvimento Científico e Tecnológico-CNPq (308569/2018-0), Fundação de Amparo à Pesquisa do Estado de São Paulo-FAPESP (2017/23776-5).

[1] Souto, Eliana B., et al. "Therapeutic interventions for countering leishmaniasis and chagas' s disease: from traditional sources to nanotechnological systems." Pathogens 8.3 (2019): 119.

Additive manufacturing of layered Fe-Si composites for soft magnetic applications: A critical review

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Better-performing electrical machines rely on the development of soft magnetic materials with fewer losses owing to hysteresis and eddy currents. Electric steels (Fe-Si) have long been used in electrical machine cores as a ferromagnetic material. Despite their inexpensive cost, Fe-Si steels with Si concentrations up to 3.2% by weight have low electrical resistivity, resulting in high eddy current losses. As the Si concentration increases, the electrical resistivity increases, and the magnetic properties are improved, namely near-zero magnetostriction, and near-zero magnetocrystalline anisotropy at 6.5 Si wt. %. However, the material becomes brittle, making the production process more complicated. To boost eddy current resistance, the core is made of thin sheets of Fe-Si steel with an insulating layer between each consecutive lamination. This geometric arrangement can be replicated by the laser powder bed fusion processing, which produces nearly completely dense metallic components layer by layer straight from computer-aided design data. This contribution critically analyzes the state-of-the-art progress concerning the fabrication of layered Fe-Si composites by additive manufacturing for soft magnetic applications. Some key topics requiring attention for further development will be highlighted and discussed.

Acknowledgments: Angelo Fernandes Andreoli - FAPESP 2021/12056-7; Vitor Eduardo Pinotti - FAPESP 2022/05528-2; Piter Gargarella - FAPESP 2021/12056-7, 2022/05528-2.

Additive manufacturing of TPU/HNT nanocomposites: Influence of rheological behavior on printing quality

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Thermoplastic polyurethanes, TPUs, are polymeric materials with elastomeric properties and processability like that of thermoplastics, being a material of choice for various biomedical applications due to their flexibility, biocompatibility, biostability and electrical insulation [1]. The use of halloysite nanotubes (HNT) as reinforcing filler in TPU nanocomposites has been studied since this nanoparticle has high aspect ratio, good mechanical properties, biocompatibility, and hollow tubular structure [2]. Additive manufacturing (AM) can be used for forming biomedical devices and, therefore, the production of 3D printed devices based on TPU/HNT presents great potential. In this work, filaments of TPU and TPU/HNT (95/5 and 90/10 w/w) were obtained by twin-screw extrusion. The influence of HNT on the thermal (DSC and TGA) and rheological behavior was measured. The filaments were molded in type IV (ASTM D638) tensile specimens by AM using a Fused Filament Fabrication (FFF) printer and the printing quality (visual inspection and dimensional stability) was correlated to the rheological properties. The presence and content of HNT did not significantly influence the thermal behavior of the TPU; however, the rheological behavior was greatly affected, since the higher the HNT content, the greater the G'' and G” at low oscillation frequencies, in addition to a significant increase in pseudoplasticity in the nanocomposites, indicating a lower viscosity and elasticity at high strain rates, typical of the 3D printing process. Despite the reduction in the printing quality of the nanocomposites when compared to TPU, a good dimensional accuracy between the printing pattern and the printed specimens was obtained.

Acknowledgements: FAPESP (2020/04937-0) and CNPq (427639/2016-6)

References:
[1] D.A. Babb, Adv. Polym. Sci. 245, 315 (2012)
[2] J. Marini et al., Polymer 55, 5226 (2014)

Analysis of characteristics and application potential of paving bricks

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In antiquity, agropastoral activities were carried out by animals and the lack of quality of transport routes made people look for ways to improve. The first reports of the use of ceramics as road coverings date back to 5000 BC. In Brazil, the first reports of the use of paving with the use of ceramic material date back to 1940, in Acre; due to the lack of raw material, such as stones, and the abundance of clay, the use of this type of pavement has become quite viable. The paving brick, also known as adoquim , is a product based on clayey mass and non-plastic materials. Depending on its production process and raw materials used, the paving brick is capable of withstanding different types of traffic, ranging from pedestrian sidewalks to roads of varying use. The research aims to evaluate, through a bibliographic review, the characteristics and the application potential of paving bricks. As a result, it is possible to see that the product has some advantages such as durability; have different surface designs or configurations; it has a fast operating time; good physical properties, as they are resistant to fuels, greases, oils and lubricants; good mechanical properties, such as high compressive strength, resistance to combined bending-traction efforts, hardness, abrasion resistance and slip resistance; low cost of maintenance and conservation [1]. With the research, it is possible to conclude that adoquim is an innovative product, still not widespread in Brazil. Because of this, the country does not have legislation or norms to standardize and guarantee their technological quality. However, countries like Spain, Colombia and the United States already have regular application of the material, framed in local norms.

References:

[1] R. S. G. Araujo. Estudo da adição de feldspato em massa cerâmica de telhas prensadaspara produção de adoquim. Dissertação (Mestrado em Engenharia de Materiais). InstitutoFederal do Piauí, Teresina, 2019.

Assessing the buildability of cementitious matriceswith chemical admixtures designed for 3D printing

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The principle of 3D printing involves the layer-by-layer construction of structures from a digital model. To determine the feasibility of using cement mixtures for 3D printing by robocasting, rheological properties in the fresh state and mechanical strength in the hardened state are crucial influencing factors [1,2]. This study presents a roadmap for evaluating the buildability of cement mixtures. Various tests were conducted on cement pastes with different water/cement ratios (ranging from 0.26 to 0.36) and chemical admixtures, including a superplasticizer (at 0.25 wt.% and 0.5 wt.%) and a setting accelerator (at 1 wt.%, 1.5 wt.%, and 2 wt.%). The tests involved overlapping molded filaments to analyze load-bearing capacity and filament deformation at different time intervals (5, 10, 15, and 20 min). The buildability was considered adequate for mixtures capable of supporting 12 layers and with a thickness variation of less than 10%. Additionally, a slump flow test was conducted to correlate with the buildability. The results revealed that mixtures without additives with water/cement (w/c) ratios ranging from 0.26 to 0.32 were buildable for time gaps of 10 min or longer. For w/c of 0.34, the buildability is achieved with time gaps of 15 min or longer. Smaller ratios were difficult to mix and larger ratios provided minimal load-bearing capacity. The chemical admixtures proved beneficial in reducing time gaps and filament deformation, especially for 1.5 wt.% or more of setting accelerator and up to 0.25% of superplasticizer. Results from the slump flow test indicated that samples with a spreading diameter below 180 mm exhibited acceptable buildability.

Acknowledgements:

Research Support Foundation of the State of Bahia (FAPESB).

References:

[1] M.T. Souza et al., Constr. Build. Mater, 314, 125666, 2022.

[2] M.T. Souza et al., J. Build. Eng., 32, 101833, 2020.

Biosilica and spongin from marine sponges for the manufacture of 3D printed scaffolds: physicochemical and morphological characteristics

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Natural materials have been studied as the best alternative for application as biomaterials due to their characteristics such as: biocompatibility, biodegradation and low cost [1]. Recently, natural materials extracted from marine sponges such biosilica (BS) and spongin (SPG) have shown positive results in tissue regeneration [2]. Moreover, 3D printing have been showing great effectiveness in producing scaffolds with natural materials [3]. So, the aim of the present study was to evaluate and compare the physicochemical and morphological characteristics of 3D printed scaffolds of BS and BS/SPG. The marine sponges Dragmacidon reticulatum and Aplysina fulva were used to BS and SPG extraction, respectively. For the manufacture of scaffolds, the Octopus™ - 3DBS bioprinter was used. The physicochemical and morphological characteristics were analyzed by FTIR, evaluation of mass loss and porosity, pH measurement and scanning electron microscopy. In the FTIR, BS peaks were similar with silica materials and SPG peaks demonstrated similarity with collagen peaks. The porosity of the scaffolds was between 44% and 90%. The mass loss rate of the BS scaffolds was between 6% and 60% while in BS/SPG scaffolds it was between 6% and 66%. In the pH measurement, the BS scaffolds maintained their pH between 5.64 and 7.18 while the BS/SPG scaffolds maintained between 5.81 and 6.10. SEM demonstrated that the BS spicules are very evident and the SPG fibers mix with them. With these results it is possible to conclude that BS and BS/SPG scaffolds have characteristics that facilitate the construction of porous scaffolds with controlled degradation. Moreover, they also maintain a pH favorable to cell growth.

Acknowledgements:
FAPESP (2021/11845-8).

References:
[1] Insuasti-Cruz, E. et al. Adv. Healthc. Mater., 11 (2022). [2] Granito, R.N.; Custódio, M.R.; Renno, A.C.M. J. Biomed. Mater. Res. Part B Appl. Biomater, 105 (2017). [3] Sousa, K.S.J. et al. Mar. Bio., (2023).

Cationic etherification modification of cassava starch: biocidal and physicochemical properties

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Starch is the most abundant biopolymer in the world after cellulose, found in roots, cereals, legumes, tubers, fruits, and seeds. The main commercial sources of starch are potatoes, corn, wheat, rice, and cassava. Cassava starch is considered a biopolymer with high potential for applications in various industrial sectors. It is present in the composition of several industrial products, but stands out in the paper and cardboard, personal hygiene, pharmaceutical, chemical and textile industries, and in the production of plastic packaging [1].

In view of the wide industrial application of cassava starch, the number of researches is increasing with the aim of providing new physicochemical characteristics to this biopolymer [2]. In this context, the cationization of cassava starch by quaternary ammonium salts, through the chemical reaction of quaternization, is shown to be an interesting and promising alternative, with the aim of conferring mainly biocidal properties to the starch.

In this study, cationic starch was prepared by etherification reaction of glycidyl-trimethylammonium chloride (GTMAC) and cassava starch. The synthed cationic cassava starch showed efficacy against SARS-CoV-2 virus and satisfactory inhibitory activity against Escherichia coli and Staphylococcus aureus bacteria, making it a new material with potential application in several biotechnological areas. The physicochemical characterization of cationic cassava starch was carried out by elemental analysis (C,H,N), Fourier-transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA), and nuclear magnetic resonance (C13 NMR).

Acknowledgments:
The authors would like to acknowledge the Coordination for the Improvement of Higher Education Personnel - CAPES (Grant 88887.513483/2020-00) and São Paulo Research Foundation - FAPESP (Grant 2021/ 09341-1) for the financial support.

Comparative investigation of the corrosion resistance of as-produced 17-4 PH stainless steel fabricated by two additive manufacturing technologies

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In this work the corrosion resistance of as-produced 17-4 PH stainless steel samples fabricated by additive manufacturing (AM) using Laser Power Bed Fusion (PBF) and Laser Directed Energy Deposition (DED) technologies were compared. In these two AM techniques pieces are produced layer-by-layer; however, whilst in PBF a high-energy laser beam scans areas previously covered by the powder producing the net-shaped pieces, in DED the laser beam is focused on a specific area, and the powder is simultaneously injected through a nozzle, resulting in different thermal history and microstructural patterns. The two producing technologies also differ by their cooling rates and geometric tolerances [1]. For the electrochemical measurements, a 3-electrode cell was used, with the samples (working electrode), the SCE (reference electrode) and a platinum spiral (auxiliary electrode) in NaCl 5% (m/m). In the experimental procedure, monitoring of the OCP was performed, consecutively, during 12h, and EIS measurements were acquired after each period (12 h and 24 h of immersion). The OCP values at 12h and 24h were: EOCP(PBF)= -0.11 /-0.07 V and EOCP(DED)= -0.13 / -0.11V, respectively. Electrical equivalent circuit fitting of the EIS diagrams did not show relevant variation between the samples. Nevertheless, potentiodynamic polarization curves after 25h showed Epit +0.41 V and +0.24V to PBF and DED, respectively, indicating a higher resistance to pitting on PBF sample. Microstructural analyzes carried out before and after the electrochemical tests showed different pitting mechanisms that are correlated with thermal stress and cooling patterns during samples' production. In PBF processed samples, pits nucleate in the centerof the cell-like grain typical of this AM technology. On the other hand, those processed byDED present pits starting along the melt tracks.

[1] A.B. Badiru, V.V. Valencia, D. Liu, Additive Manufacturing Handbook, CRC Press, 2017. https://doi.org/10.1201/9781315119106.

CONCRETE BLOCKS SUBMITTED TO BALLISTIC IMPACT TESTS: REINFORCEMENT PERFORMANCEAND IDENTIFICATION OF LOCAL AND GLOBAL DAMAGE

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Concrete blocks subjected to ballistic impact testing: Reinforcement performance and identification of local and global damages Wesley Machareth Lisboa1,2, Diego Pinheiro Sodré1,2, Fernanda Gomes Pixioline1,2, Joaquim Teixeira de Assis1,2, Joel Sanchez1,2 1Universidade do Estado do Rio de Janeiro, 2Instituto Politécnico de Engenharia e-mail: wesley.m@pm.rj.gov.br, diego.p@pm.rj.gov.br, fernanda.g@pm.rj.gov.br, Joaquim.iprj@gmail.com, jsdominguez@iprj.uerj.br Statistics published by Public Security Agencies justify the increasing demand for construction methods that enhance the sense of security [1], marking recent years with a significant growth in investments in protection systems. Concrete, in turn, proves to be an important tool for developing products with this purpose and shows promising results when subjected to ballistic impact. This study presents the performance of concrete samples, with and without internal reinforcement, with an average compressive strength of 40 MPa, subjected to ballistic impact caused by projectiles of caliber 0.40", 5.56 mm, and 7.62 mm. For the experiment, three concrete samples were produced, one with internal reinforcement of aramid fiber, another with internal reinforcement of 1020 steel, and another without internal reinforcement. All samples received the Sika® Concrete Forte plasticizer as an additive. The specimens were producedand classified according to adaptations of ABNT/NBR 15000:2020 and ABNT/NBR 5983:2015 standards. The samples were subjected to impacts fired at a distance of fifteenmeters. As the tests were carried out, the occurrence of local damages, such as the formation of craters and tunnels, and global damages, such as crack propagation and material detachment on the impact faces of the samples, were verified [2]. Fragmentation on the opposite face of the impact was also observed in one of the samples. These resultsdemonstrate that concrete, even without reinforcement, exhibits good re

DEFINITION OF PROCESS PARAMETERS BY DIRECT ENERGY DEPOSITION (DED) FOR AISI 431L STAINLESS STEEL GAS TURBINE BLADES

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The objective of this study was to define a process map for the growth of gas turbine blades using direct energy deposition. For additive manufacturing, AISI 431 L stainless steel powder was used, and the blades were grown on an AISI 304 L steel substrate. In gas turbines, the blades are composed of alloys resistant to high temperatures and are considered to have the highest specific cost [1]. The blades are subjected to a corrosive environment, at high rotation and high temperatures [2]. DED depositions were carried out using BeAM modulo 250, equipped with linear motors on five axes. The process control was carried out using Siemens 840D equipment, while the laser source was a Yb:glass, with 1kW of power, from IPG Photonics. The powder sprinkler system (head) and powder distributor belong to BeAM. Thirty-six parameters were reproduced with only one bead, and the process variables were scanning speed from 1800 mm/min to 2200 mm/min, power from 300w to 550w, and powder mass flow from 4,7g/min and 8,42g/min. After performing the thirty-six parameters, the six best parameters were chosen to be reproduced, considering the best adhesion between bead and the substrate, a more homogeneous bead, and one that promotes complete fusion of the bead. Through parameterization, it was possible to grow the blade by LP-DED, without limiting the number of layers. From the preliminary analyses, the parameters used to produce Six different supersonic airfoils were: 2000 mm/min speed, 450 W powerand 8.42 g/min powder mass flow. The choice of these parameters is due to the volumetric defects and hot cracks between the layers.

Acknowledgment:

This research was carried out with the support CAPES and FAPESP.

References:

[1]SIVAKUMAR, R., MORDIKE, B. (1989).high temperature coatings for gas turbineblades: A review. Surface and Coatings Technology, pp. 139-160. [2]PADTURE, N., GELL, M., JORDAN, E. (April 12, 2002).Thermal BarrierCoatings for Gas-Turbine Engine Applications.

Development of model dispersed systems to obtain Total Oil and Grease estimates in apparatus based onacoustic properties and 3D printing

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Produced water (PW) in oil production operations contains both dispersed and emulsified oil. The Total Oil and Grease (TOG) in water is a parameter controlled daily for disposal or reinjection, and have limits stipulated by environmental agencies. The reference method accept by IBAMA is made through the partition-gravimetric, which is very susceptible to quantitative errors and not practical. To control the quality of PW on oil platforms, different benchtop analysers are used for TOG measurements. Studies related to new technologies concerning fluid monitoring are important to industry and acoustic techniques can be likely succeed for this purpose. The aim of this work was to develop model dispersed systems, simulating PW, with known and controlled physical-chemical properties, in order to facilitate the development of an assertive methodology based on acoustic properties to estimate TOG values. Concentrated emulsions of oil (hexadecane) in water (O/W) were prepared in varying proportions of O and W with a mixture of non-ionic surfactants. From there, dispersed systems with low oil content (oil range from 5 to 1,000ppm) were obtained. All systems were characterized in terms of visual stability, viscosity and distribution and size of the drops as a function of time. The correlation between acoustic properties and TOG is made through transducers coupled to an apparatus built by 3D printing. The results showed assertiveness in the proposed method and in the choice of constituents for the stable and reproducible dispersed system preparation. Acknowledgments We thank Petrobras and ANP for the financial support. References [1] Silva, L. Desenvolvimento de uma técnica ultrassônica para avaliar teores de óleo e graxa em efluentes de biocombustíveis, Química nova, vol 38, 2015.[2] Al-Ghouti. M, Al-Kaabi. M, et. al. Produced water characteristics, treatment, and reuse: A review. Journal of Water Process Engineering, vol 28, 2019.

Dilution and Solidification Pattern in H13 Tool Steel Deposition Over a Microalloyed Steel Substrate Through Direct Energy Deposition (DED)

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H13 tool steel is used to produce dies and molds due to properties as wear resistance and toughness, stable under high temperature [1, 2]. Traditionally, they are produced through several machining steps, followed by heat treatments to control microstructure and achieve desired properties [1]. Direct Energy Deposition (DED) additive manufacturing (AM) is gaining considerable attention in tooling fabrication due to its capability to produce customized geometries, complex cooling systems and functionally graded microstructures [1, 2]. DED process parameters are critical to the final properties [1]. Therefore, the purpose of this study was to evaluate the number of layers influence in depth penetration and geometrical dilution with fixed energy density, through cross-section observation, using optical and scanning electron microscopy, and microhardness analysis. The present work used H13 steel powder, water atomized in a Brazilian company, deposited through DED/AM over a microalloyed hot rolled steel sheet, generating a stack of layers, whose overlap involved an in-layer post heating [2], remelting part of the upper tracks and heat treating the bottom ones [1]. This resulted in columnar grains morphology close to substrate due to epitaxial growth, and reduced columnar grains length with changed microhardness values in re-heated areas at layers intersections, as expected [2]. Finally, despite some observed voids, it is concluded that the deposition was adequate for DED process first stage anchoring the H13 steel deposit in the substrate as function of the suitable geometrical and chemical dilution depth at first layer-substrate interface [3].

Acknowledgements:
FINEP, CAPES (Pró-Defesa IV and PROEX)

References:

[1] ZHAO, X. et al., Materials Today Communications 25, 101390, (2020).
[2] JOSHI, S. et al., Journal of Manufacturing Processes 68, 852-866, (2021).
[3] PELLLIZZARI, M. et al., Surface & Coatings Technology 432, 128084, (2022).

Effect of intercalated layer thickness on the mechanical properties of CP Ti/Ti-6Al-4V composites produced by laser powder bed fusion technique

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Recent advancements in additive manufacturing allowed the development of titanium parts with promising mechanical properties and complex geometries. The process also allows for controlled material distribution, which can be utilized to produce composite materials with distinct characteristics, resulting in a product with superior properties. This study investigates the reinforcement effect of adding intercalated layers of varying thicknesses (150 µm, 300 µm, and 450 µm) of Ti-6Al-4V alloy to a CP Ti matrix with a fixed thickness of300 µm. The samples were produced by the laser powder bed fusion (L-PBF) technique using a Yb:YAG laser with a power of 209 W, combined with a scanning speed of 700 mm/s. The microstructural analysis of the composites was performed through optical microscopy, scanning electron microscopy, and energy dispersive spectroscopy. The mechanical properties were evaluated by Vickers microhardness and compression tests. The high cooling rates lead to α' phase provoked by a martensitic transformation of the β phase. CP Ti layers have a Vickers hardness of approximately 255 HV, while the average Vickers hardness of the Ti-6Al-4V layers was found to be 350 HV. Compression tests showed a yield strength of about 1000 MPa, allied to a strain of about 45%. The results suggested the possibility of multi-material processing, tailoring the properties of the products according to the layer thickness.

Acknowledgments:

The authors acknowledge the São Paulo State Research Foundation (FAPESP), grant #2018/18293-8 for its financial support.

Effects heat treatment on microstructure and mechanical behavior of an additively manufactured Hybrid Ti alloy

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Recently, the manufacturing of Ti alloys has seen significant evolution, thanks to the evolutions in additive manufacturing (AM) technology. This approach now allows for the creation of a vast array of products with intricate shapes and compositions, by applying a layer-by-layer deposition method. One of the most efficient AM techniques is laser powder bed fusion (L-PBF). It selectively melts metallic powder with a laser beam source. In general, the feedstock processed by L-PBF corresponds to pre-alloyed powders. If different powders with different mechanical behaviors are mixed and used as feedstock, a hybrid microstructure can be achieved [1]. The mixed powders can produce unique microstructure features, such as the formation of micro-regions rich in a particular component within a distinct matrix. In this work, a mixture of two different powders, corresponding to the ductile Ti-42Nb (wt%) alloy and the high mechanical strength Ti-5553 alloy, was utilized as feedstock. They were mixed in an eccentric rotary mixer. The samples were printed on an OmniSint-160 machine equipped with Yb:YAG fiber laser. The obtained samples were heat treated to produce regions with different mechanical features and characterized using scanning electron microscopy, X-ray diffraction, differential scanning calorimetry and nanoindentation tests. The results obtained suggested the formation of compositional microregions in the printed material, which led to heterogenous mechanical behavior after the aging heat treatment.

Acknowledgements:

The authors acknowledge the São Paulo Research Foundation (FAPESP), grant #2018/18293-8 and #2022/10049-6, for its financial support.

References:

[1] A. Zafari, K. Xia, Superior titanium from hybridised microstructures - A new strategy for future alloys, Scr Mater. 173 (2019) 61-65.

Enzymatic oxidation of lignosulfonates for aldehyde-free adhesive formulation

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New products obtained from biomass biorefining can become an economically viable and environmentally sustainable reality. Product diversification in the biorefinery portfolio, including lignosulfonates and oligosaccharides, besides glucose and xylose can contribute to the techno-economic viability of biorefineries. As lignosulfonates are phenolic compounds, enzymatic oxidation can increase their molecular mass adding value to the final products. The present study aimed to evaluate the laccase-initiated oxidation of lignosulfonates from different plant materials (conifers and grasses), allowing application as an aldehyde-free adherent matrix for wood fibers. Enzymatic oxidation systems of lignosulfonates, both from conifers and sugar cane, using Trametes versicolor laccase demonstrated an increase in the molar mass of the treated lignosulfonates. For conifers, the molar mass increased from 1.9 kDa to 5.4 kDa, while the oxidation of sugar cane lignosulfonate increased from 1.1 kDa to two majors products with increased molar masses of 22.2 kDa and 2.8 kDa. This reaction system has been explored in fibers adhesion studies. Enzymatic oxidation of eucalyptus fibers promoted adhesion via oxidative crosslink of lignin residues. Unexpectedly, the presence of lignosulfonates, both from conifer and sugarcane bagasse, reduced the adhesion effect, most likely due to the solubility of the enzyme and the lignosulfonate having driven the radical coupling reactions in the aqueous medium and not on the fiber surfaces. This fact is evident when observing a profile of molar mass increase of the lignosulfonate collected in the washing water of the adhered fiber, being 3.2-fold for conifer lignosulfonate and 2.5-fold and 14.3-fold for sugarcane lignosulfonate. This adversity is being overcome by studying reaction systems with low moisture content forcing polymerization reactions to occur at the fiber surfaces.

Evaluation of the influence of different molecular weight polydimethylsiloxanes on the aggregation ofasphaltenes by atomic force microscopy

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During oil production, undesirable foam formation occurs, which is stabilized by surfactants naturally present in oil, called asphaltenes. Currently, to remedy the problem of foam, polydimethylsiloxanes (PDMS) are used [1]. Therefore, the present work aims to evaluate the performance of PDMS of different molar masses in the formation of asphaltenes agglomerates using atomic force microscopy (AFM). As a result of the AFM images, it was possible to observe that PDMS interferes in the packing of asphaltenes, showing that the efficiency of these PDMS as defoamers is directly linked to the action of these products in the agglomeration of asphaltenes at the oil/air interface.

Acknowledgment:
To Cenpes/Petrobras, CNPq, Capes e Faperj.

Reference:
[1] Cevada E, Hernández E, Flores C, Zavala G, Álvarez F, Vázquez F. N. Fuel, V. 278:118401 (2020).

Exploring the Use of Metallic Binder in Additive Manufacturing of Nd-Fe-B Magnets: Improving Density and Magnetic Properties

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Additive manufacturing (AM) of composite magnets using Laser Powder Bed Fusion (L-PBF) has shown great potential for achieving complex geometries and custom designs with high precision [1]. However, the porosity and magnetic properties of the final magnets are highly dependent on the processing parameters and feedstock characteristics [2, 3]. In this study, we explored the replacement of the polymeric matrix of the composite matrix with a metallic binder to improve density and magnetic properties. The feedstock, comprising Nd-Fe-B powder and polyamide-12 powder, was laser processed into low-density bonded magnets, which were then subjected to a sintering heat treatment. A systematic study was carried out to evaluate the change in magnetic properties of composite Nd-Fe-B magnets obtained using L-PBF. It was observed that the use of a rare-earth-based alloy metallic binder and the subsequent sintering heat treatment substantially increased the coercivity values from around 50 kA/m to more than 500 kA/m. This was due to the microstructural interactions between the infiltrated metallic phase and the ferromagnetic particles, altering the coercivity-induced mechanisms. This study sheds light on how microstructural changes introduced after the L-PBF process can improve magnetic properties, opening up new avenues for the design and development of high-performance magnetic materials for additive manufacturing.

[1] CHAUDHARY, V. et al. Progress in Materials Science v. 114 p. 100688 (2020)
[2] Engerroff, J.A.B. et al. Journal of Rare Earths. v. 37, p. 1078 (2019)
[3] FIM, R.G.T. et al. Additive Manufacturing, v. 35, p. 101353 (2020)

Filaments of polycaprolactone/ sodium alendronate and chitosan - a supplier for additive manufacture

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The destruction of bone microarchitecture characterizes osteoporosis, loss of mass, decreased bone strength, and increased risk of fractures. Bisphosphonates, for example, sodium alendronate (ALD), are usually applied to treat osteoporosis [1]. ALD presents some collateral effects in oral and intravenous administration. Among them, it is possible mention: irritation of the esophagus, fevers, ocular inflammation, and mandibular osteonecrosis. Polymeric materials play an essential role in biomaterial production for bone tissue regeneration. Thus, combining the appropriate polymer with drugs is critical to producing a suitable drug delivery system. Polycaprolactone (PCL), a biocompatible polymer, has been used in additive manufacturing techniques [1]. Chitosan (Chi) is a polysaccharide widely studied for drug delivery applications. In this work the hot melting extrusion (HME) technique was applied to produce PCL/Chi/ALD filaments. This filament was analyzed by scanning electron microscopy (SEM) and Fourier infrared spectroscopy (FTIR). SEM images characteristics of all materials present in the samples. FTIR analysis shows bands related to PCL, Chi, and ALD, showing that the material's chemical properties were unaffected and no reaction occurred. The ALD release was studied by UV-Vis to evaluate the filament's ability to deliver ALD. The study was done for eight hours in phosphate buffer at 37oC. According to UV-Vis analysis, the amount of ALD delivery was 1,04mg/cm of filaments, equivalent to 5.24mg of ALD/ god filaments. These results indicate that this filament is a good supplier for producing biomaterials for drug delivery using additive manufacturing, allowing the obtention of personalized devices.

Acknowledgments: To the CAPES (Financing Code 001); CNPQ) and FAPERJ (E-26/010.000983/2019).

References:

[1] da Silva, T.G, Pereira, D. B Patricio, B.F.C., Sarcinelli, M.A., Rocha, H. V. A., Letichevsky, S., Mendonça, R. H. J. Appl. Polym. Sci , 2021, 138(28), 50678.

Fracture toughness of Ti42Nb and Ti5553 sandwich structure produced by Laser Powder Bed Fusion

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The aerospace industry often needs new materials with high strength and low density, but these are usually expensive and might have limitations in their applications due to low fracture toughness.[1] Thus, the aim of this study was the development of sandwich structures composed of different Ti alloys: Ti-42Nb and Ti5553 (Ti-5Al-5V-5Mo-3Cr-0.5Fe) to improve fracture toughness. This approach involved using layers of Ti42Nb with 150 μm thickness and Ti5553 with 300 μm processed by Laser Powder Bed Fusion.[2] The results indicated improvement in fracture toughness while satisfactorily maintaining the low density and with little influence of porosity due to the manufacturing process of differentlayered materials.

AcknowledgmentsThis work was supported by CAPES and the São Paulo Research Foundation (FAPESP), Grant # 18/18293-8.

References[1] Schwab, H., et al. Microstructure and mechanical properties of the near-beta titanium alloy Ti-5553 processed by selective laser melting. Materials Design 105 (2016): 75-80.[2] Huang, Sheng, et al. Fracture behavior of laser powder bed fusion fabricated Ti41Nb via in-situ alloying. Acta Materialia 225 (2022): 117593.

Gas Atomization of Fe-6.5Si Powders for Additive Manufacturing: Production and Characterization

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There is a growing need for efficient and cost-effective electric motors that consume less energy [1]. Laser Powder Bed Fusion (L-PBF) has emerged as a viable alternative for processing electric steels with 6.5%wt. of silicon, as conventional techniques result in brittle behavior due to the formation of B2 and DO3 phases [1]. Gas atomization (GA) is the main method for producing metallic powders suitable for L-PBF [3]. However, powders must exhibit specific characteristics and properties, such as a Gaussian particle size distribution, spherical morphology, flowability, and high apparent density [2]. Powders of Fe-6.5Si alloy (%wt.) were characterized to validate their use in future L-PBF studies, including chemical composition, particle size distribution, morphology, flowability, apparent density, and microstructural aspects to evaluate the presence of brittle phases. (Acknowledgments:Vitor Eduardo Pinotti - FAPESP n° 2022/05528-2; Angelo Fernandes Andreoli - Projeto FAPESPn° 2021/12056-7; Mayumi Andrade Nakahashi - FAPESP n° 2022/12178-8; Piter Gargarella - FAPESP n° 2017/27031-4 and CNPq n° 405545/2021-5. Federal University of Sao Carlos, Graduate Program in Materials Science and Engineering. This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.)

Heat Treatment of Quasicrystalline Phase Forming Al95Fe2Cr2Ti1 Alloy Produced by Additive Manufacturing

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Most high-strength aluminum alloys obtain this property after precipitation-hardening heat treatments, a strategy used for over 100 years [1]. The number of alloys where this strategy can be applied is limited by the low number of elements that have significant solubility in aluminum. Another possibility to strengthen aluminum alloys is by forming quasicrystalline phases [1]. Recent data have shown that it is possible to obtain a metastable quasicrystalline phase in a large volume of Al-Fe-Cr-Ti alloys manufactured by Laser Powder Bed Fusion (L-PBF) [2]. These alloys are promising in applications such as matrices and structural parts, as they have high mechanical strength, especially for applications at high temperatures. These alloys are expected to exhibit high fracture toughness due to a microstructure that combines the presence of a high-strength quasicrystalline phase in a highly refined ductile aluminum dendritic matrix due to the high cooling rates involved in the L-PBF process (10^5 K/s) [2]. The precipitation heat treatment of these Al-Fe-Cr-Ti alloys manufactured by L-PBF was not studied so far. Thus, this project consisted of carrying out a series of heat treatments of the Al95Fe2Cr2Ti1 alloy produced by L-PBF and evaluating their influence on the phase formation and mechanical properties. The results indicated an increase in hardness for certain heat treatment conditions, thus indicating that, these alloys can be applied and classified as solid solution hardened.

Acknowledgments:
Erlifas Rocha - FAPESP - Processo: n° 2022/15435-1
Guilherme Perin - CNPQ - Processo: n° 145083/2022-5Piter
Gargarella - FAPESP: 2017/27031-4

References:
[1] GALANO, M. Novel Al based nanoquasicrystalline alloys. Progress in Materials Science, 123, September, 2022.
[2] ARAUJO, A. Processability of recycled quasicrystalline Al-Fe-Cr-Ti composites by selective laser melting. A statistical approach. Materialia, 22, October, 2022.

Identification of Critical Parameters in Semi-Solid Extrusion-Based 3D Printing Process using Quality by Design Approach

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Tuberculosis constitutes a public health issue, particularly concerning arduous pediatric treatment. 3D printing (3DP), enables customized, palatable formulations with multiple drugs in a single medicine. Semi-Solid Extrusion prints medicines using a semi-solid intermediate production formulation [1]. The systematic approach begins with predefined objectives, utilizing the Quality by Design (QbD) principle to identify critical quality attributes (CQAs), critical process parameters (CPPs), and critical material attributes (CMAs) that affect the quality target product profile (QTPP). It helps to achieve high-qualityprototypes with reduced variability [2]. This study aims to identify the CPPs that impactsQTPP, defined as a gummy of isoniazid content for the treatment of tuberculosis, using a QbD approach. QTPP and CQAs were defined to identify the CMAs and CPPs. Potential risksimpacting QTPP during formulation and printing were identified through brainstormingusing Ishikawa cause and effect analysis. These risks were further analyzed using FailureMode and Effect Analysis (FMEA), considering Severity (S), Occurrence (O), and Detection (D) on a numerical scale (RPN = SxOxD). Finally, variables with the highest risk were selected based on high-risk attributes (RPN > 250). Risk analysis of CQAs using Ishikawa identified parameters with significant material impact. Six parameters and their independent variables were determined: Formulation, Rheology, Computational design, Printing process, pharmaceutical form and Packaging. Based on FMEA analysis, the high-risk CQAs are Print hole; Temperature; Formulation viscosity; Flow rate. With the high-risk CQAs defined, it is possible to conduct process improvement strategies to enhance the printing and medicine performance.

Acknowledgements:

CAPES, CNPq

References:

[1] E. Díaz-Torres1 and L .Rodríguez-Pombo2, Int. J. Pharm., Vol. 4, 2022. [2] International Council for Harmonization (ICH). Pharm. Dev. Q8 (R2), Aug. 2009.

Influence of Powder Characteristics on The Printabilityof Ceramics Inks by Direct Ink Writing

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The extrudability of an ink is composed by the flow stress and the flow behavior index. The first parameter represents the minimum shear stress required to make the ink flow, which is related to the minimum force required by the 3D-printer to extrude an ink, and the second one describes the flow behavior of the material. The shape fidelity. It is important to note that, unlike when using polymeric materials, printing is not the final step when manufacturing ceramic parts by DIW. Most ceramic inks require post-printing treatments, which involve shrinkage, very significant in the case of sintering. These dimensional changes must be taken into account when designing the CAD model of the green parts, and the shape fidelity of the green part is a crucial aspect to ensure the accuracy of the whole process.

In this study were evaluated the influence of particle , particle distribution. pH and surface area of an s alumina, zirconia and alumina-zirconia ceramics on the rheological behavior of inks employing polyvinyl alcohol and polyethilene glycol used as viscosifying and coagulant agents in deionized water. 3D printing was performed using a DURAPRINTER E02 model and was possible to obtain ceramic pieces of complex shapes with dimensional accuracy and theoretical densities up to 96%.

References

[1] - G. Gillispie, et al., Assessment methodologies for extrusion-based bioink printability, Biofabrication 12 (2) (2020).

Influence of process atmosphere in the processabilityof additively manufactured stainless steel

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Additive manufacturing (AM) is a new processing technology that involves layer-by-layer construction of a part. Laser beam powder bed fusion (PBF-LB) is one of the most applied AM processing for metals. PBF-LB involves more than one hundred parameters, where the most influential are the power and speed of the energy source. In most cases, inert atmospheres are often neglected in processing optimization. The current work evaluated the influence of two process atmospheres (argon or nitrogen) in the processability of AISI 316 L stainless steel. The type of gas in the process chamber displaces the high-density conditions. Crystallographic orientation, nitrogen content, grain size distribution, and thermal properties of bulk parts were modified due to the atmosphere applied. This work was supported by CNPq, CAPES, and FAPESP.

This research used resources of the LNNano/CNPEM (SEM facilities). The authors thank Ominitek for providing access to the PBF-LB machine, Prof. Rubens Caram for providing access to the oxygen/nitrogen analyzer.

Influence of texturing parameters on surfacemodification of UNS S32101 duplex steel

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The application of laser texturing aims to modify the surface without changing its chemical composition. These alterations can be evaluated through mechanical, physical or chemical tests. Texturing can be applied to improve efficiency in industrial processes, such as the flowability of materials, by applying laser texturing to the internal surfaces of components. This work aims to evaluate the influence of texturing parameters in the flow process of industrial batchers. UNS S32101 lean duplex stainless steel (LDSS) samples were used for the tests. For laser texturing of the surfaces, laser of Yb was used, with a wavelength of 1064 nm (infrared), using the scanning speed of 140 mm/s and pulse duration of 150 ns. Two sets of textured samples were produced: with a frequency of 50 kHz and 2 kHz. In both frequencys, the power(%) was set at 100, 60 and 20. The effect of texturing was analyzed through microstructural characterization with tests of contact angle, roughness and scanning electron microscopy.

Influence of the additive manufacturing parameters on the Ti6Al4V microstructure

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The 3D printing on an industrial scale describe the one more advanced techniques. In the manufacturing process technology the model is created by successively layering material from a digital 3D model. The 3D manufacturing process allows obtaining complex geometry, weight reduction, and customized components. A careful microstructure characterization of the produced components is necessary, since the parameters influence in surface roughness, microhardness and corrosion resistance. Samples of Ti6Al4V built with the same laser speed, hatch distance and layer thickness but different laser power were analyzed. The variation in laser power influenced the roughness and corrosion resistance of the samples.

Investigaten of 316L stainless steel parts produced by Wire Arc Additive Manufacturing - WAAM

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Additive manufacturing technologies have been revolutionizing the paradigm of manufacturing and engineering areas, due to the possibility of creating three-dimensional, complex and near net shape parts. Among these technologies, Wire Arc Additive Manufacturing (WAAM) is highlighted due to its high deposition rate, low equipment cost, high material usage (little waste) and consequent respect for the environment.

In this work, parts of austenitic stainless steel 316L were produced by WAAM with layer overlap angles of 45° and 90°, using values of U=21.2V, I=220A and WS=15mm/s, these welding parameters were defined in initial tests.

XRD measurements were performed along the Z axis´surface, on the samples´surface in order to evaluate the distribution of phases and residual stress by the sin2(phi) method, and the microstructure of the cross section of selected pieces were analyzed by SEM.

In initial analyses, a better surface quality of the sample was observed with an overlapping angle of 90°.

The majority phase present in the pieces is γ austenite, but δ ferrite was also observed, which was confirmed by XRD and SEM analyses. The ferrite presented vermicular, columnarand lathy morphologies in different regions of the samples in the SEM images.

In the XRD measurements, there is a great variation in the relative intensity of the peaks, mainly in those referring to the planes (111) and (200) of the austenite, indicating a high texture, which is characteristic of the materials produced by WAAM [2].

The part that was built with an overlapping angle of 90° preferably presented compressive stresses along the Z axis, with magnitudes between 100 and 200MPa.

Investigation of the influence of patterns and percentages of fills on the mechanical properties of 3Dprinted PLA

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The Fusion and Deposition Modeling (FDM) technology is a manufacturing method that uses polymer/filaments as a raw material. In this technique, the filament is usually heated to its molten state and then extruded through the nozzle of the 3D printer [2]. Due to the melting and deposition of the material under the bed, there are limitations of inputs that can be employed, usually, polymers such as PLA and ABS are used as they have satisfactory thermal properties [3]. This study will investigate the influence of fill patterns (hexagonal, triangular and concentric) in the mechanical properties of PLA 3D Printed The material will be Made througg Additive Manufacturing, by The FDM technique. And, characterized by X-ray Diffraction, and by Raman spectroscopy. The mechanical tensile properties of the printed polymer shall be determined in accordance with the standard [1]. The result of the XRD characterization shows that PLA has a semicrystalline nature. With regard to Raman characterization, the results show C-O-C and CH3 vibrations at 317 cm-1, C-CO vibrations at 407 cm-1, stretching of C=O at 740 cm-1, C-COO vibrations at 872 cm-1, stretching C-H3 at 1043 cm-1, vibrations of CH3 assigned in 1128 cm-1, deformation CH in 1302 cm-1, deformation CH3 in 1383 cm-1, symmetrical deformation of CH3 at 1453 cm-1, stretching of CH/C=O at 1764 cm-1, symmetrical and asymmetric stretching of CH3 at 2878, 2948, 3004 cm-1. The mechanical characterization of traction shows a direct influence of the filling pattern on the properties of the printed parts. the concentric pattern being the best result with 41.26 MPa, the hexagonal pattern presented 28.12 MPa and, finally, the triangular filling presented a limit of 11.63 MPa.

Acknowledgements:
Thanks to CAPES and Finep for financial support.

Reference

[1] ASTM D638-22, ASTM International (2022)
[1] F. M. Mwema et al., Fused Deposition Modelling, p. 1-15 (2020)
[2] M. Subramaniyan et al., Materials Today: Proceedings vol. 45, p. 820-827 (2020)

Laser beam powder bed fusion of AlSi10Mg: influenceof heat treatment on hardness and microstructure

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One of the techniques used in the Additive Manufacturing processing of aluminium alloys is Powder Bed Fusion Laser Beam (PBF-LB). As a result of laser processing, the solidification conditions lead to microstructures that are far from equilibrium, exhibiting extreme microstructural refinement and high residual stresses. The cells in AlSi10Mg samples produced by PBF-LB comprise an aluminium matrix and a continuous fibrous eutectic silicon network [1,2]. Post-processing heat treatments are typically applied to specimens produced by Additive Manufacturing to modify the as-built (AB) microstructure and subsequently improve their mechanical properties [1,2,3]. In this study five different heat treatments were studied, stress relief at 300 °C for 2 h, homogenization at 540 °C for 2 h followed by artificial aging at 170 °C for 8 h, homogenization at 540 °C for 6 h followed by artificial aging at 170 °C for 14 h, direct artificial aging at 155 °C for 6 h and direct artificial aging at170 °C for 2 h. The as-build microstructure showed a typical PBF-LB cellular-dendritic solidification structure, that following direct artificial aging the silicon network undergoes partial degeneration, and the solidification structure was erased after stress relief, homogenization followed artificial aging heat treatments. The hardness value of the AB was 133,6 ± 3,9 HV, while that of the direct artificial aging treatment samples was 153,5 ± 4,2HV. On the other hand, the other heat treatments decrease the hardness compared to theAB condition.

Acknowledgements:
The authors thank LNNano/CNPEM for providing access to the SEM facilities, Prof. Rubens Caram Junior for providing access to the nanoindentation equipment, and Fundings agencies CNPq, FAPESP and CAPES.

References:
[1] Araújo, L. C. et al. International journal of mechanical sciences, v.213, 2022. [2] Yaru, L.et al. Materials science and technology, 2023. [3] Giovanola, M. et al. Materials science and engineering a, v.802, 2021.

Laser parameters, powder geometry and the influenceon roughness of Ti-13%Nb-13%Zr alloys produced by Laser Powder Bed Fusion

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L-PBF is capable of creating complex and customized products quickly and precisely by depositing molten powders using a laser onto layers. The Ti-13%Nb-13%Zr alloy has excellent properties in terms of corrosion resistance, low elastic modulus and high biocompatibility, making it a great possibility for applications as biomaterial. The optical perfilometry technique simultaneously illuminates the surface studied and a reference surface. When the light is reflected by both surfaces, a topographical representation is formed, characterizing the roughness of the metal surface. The morphology of the powders can be decisive during the manufacture of products by L-PBF, and for this reason, this work investigated the influence of spherical and irregular powders in the additive manufacturing of parts using different powers, since the research of low roughness can avoid later surface treatments. HDH samples showed a slight increase on the number of spatters (superficial defects) comparing to the PA samples. Nevertheless, the difference was not very significative when assumed the same laser power. A microscopic analysis shows that the metal roughness is associated to oblique lines of the surface. These lines possibly correspond to solidification fronts created during the L-PBF process. For lower laser powers, these lines can be presented as very clear "V" shapes called "ripples", pointing tothe opposite direction of the laser movement. For neighboring tracks, these ripples point toopposite directions between themselves. It´s possible to conclude that bigger laser powersresult in more parallel ripples, hiding part of themselves by the neighboring tracks. By perfilometry was possibly to infer that: A power of 200W resulted in bigger roughness whenfabricated products made of HDH powder; The lowest roughness was obtained by PA powder and 250W and it is also possible to conclude that roughness is inverselyproportional to the laser power for all samples.

Microstructural and mechanical characterization of the maraging 300-grade steel processed by additive manufacturing (L-PBF) using different powder feedstock

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The production of parts by additive manufacturing (AM) has grown in recent years, mainly due to decreased cost and the insertion of new materials for applications taking advantage of the complex geometries that can be fabricated. Maraging steels are ferrous alloys presenting ultra-high strength. Maraging 300-grade steel with 18% nickel (18Ni300M) has been adopted for applications that demand high mechanical strength and dimensional stability, such as in the aerospace and military fields [1]. Recent works investigated the mechanical and microstructural properties of 18Ni300M steel processed by L-PBF [1]. However, studies on the particulate raw material's influence on the parts' properties are scarce [2]. The main objective of the present work is to investigate the microstructures of two feedstock powders of 18Ni300M steel (X and Y) and evaluate the mechanical properties of LPBF parts manufactured from these powders. The powder X was gas-atomized by an AM commercial provider and Y was water-atomized by a local atomization company. The material processing by L-PBF was performed at FEQ/UNICAMP. Microstructural and mechanical characterizations were conducted at EEL/USP. From the results, it was verified that particle distributions of the two feedstock powders diverged concerning the data provided by the atomization companies. In addition, powder Y presented worse morphometric parameters than powder X, which compromises the L-PBF process, as evidenced by the porosity in the built samples. Due to these defects, samples processed withbatch Y exhibited a lower mechanical response, particularly in ductility (11% for batch Xand only 4% for batch Y). Furthermore, fractographic analyses revealed defects such as pores and inclusions in both materials. However, the defects in batch Y appeared to be moresevere and could compromise the material's structural integrity.

References:

[1] L. Guo, et al., vol. 120, p. 227-252 (2022).
[2] F. Deirmina, et al., vol. 24, p. 1-13 (2022).

Microstructural Characterization of the High Strength AA2017 Alloy Produced by Laser Powder Bed Fusion

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High Strength Al-Cu alloys are widely applied in the aerospace and automobile industries due to their high specific strength, and good fatigue and corrosion resistance. Nevertheless, the processing of Al-Cu alloys by Laser Powder Bed Fusion (L-PBF) remains a challenge because of their large solidification interval, which leads to hot cracking in the final piece. Based on this, the objective of this work was to process the AA2017 alloy, which is the oldest and best known alloy of the Al-Cu system, by L-PBF. Box Behnken design of experiments (DoE) and analysis of variance (ANOVA) were used to assess the influence of the processing parameters (i.e. laser power, scanning speed, and hatch distance) on residual porosity and relative density. Optical microscopy and scanning electron microscopy equipped with an energy-dispersive X-ray spectrometer were used to characterize the microstructure of the alloy processed by L-PBF. The set of process parameters optimized via DoE/ANOVA resulted in high relative density (>99%) and low residual porosity. The as-built microstructure was characterized by the typical α-Al columnar grains along the building direction and Al_2Cu precipitates. Precipitates with a concentration of Mg and Si, and Fe and Mn were also observed and identified comparing with the phases formed in the solidification of the alloy according to Thermocalc computational thermodynamics software (version 2021b), TCAL3 database, and the literature.

Acknowledgments:

The authors thank the grants #2020/09544-7, #2022/02760-1, and #2017/27031-4, São Paulo Research Foundation (FAPESP). This study was financed in part by the Coordenaçãode Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001 andThe Austrian aviation program ‘ ‘TAKEOFF” (PILOT, grant number 852796, 2018) and BMK - The Austrian Ministry for Climate Action, Environment, Energy, Mobility, Innovation and Technology.

Modulating alginate-based hydrogels crosslink densityvia calcium ions

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Hydrogels are a type of polymer-based network with ability to absorb water and other bodily fluids and capacity to mimic the extracellular matrix. Because of this unique property, hydrogels are widely used in tissue engineering through a versatile platform after rational structure and functional design that leverages on material engineering to modulate its physicochemical properties, and influence cell signaling cascades and fate [1]. Alginate is a natural polymer that has garnered significant attention due to its biocompatibility and ability to crosslink in a fast and efficient manner. One of the key advantages of alginate-based hydrogels is their ability to undergo rapid crosslinking through the attachment of carboxylic groups to a divalent metal ion during their formation. Calcium is the most commonly used metal ion for alginate gelation, as it allows for a rapid gelification and is also an essential mineral for human metabolism. However, the hydrogel resulting from this mixture is highly sensitive to the conditions under which it is prepared. Here, we investigated the effect of Ca2+ on the structural and rheological properties of alginate hydrogels, aiming to obtain gels with high homogeneity and stability. FTIR was used for the structural characterization of the hydrogels, while the water content and swelling degree of hydrogels were determined using the gravimetric method. Our findings revealed a strong influence of Ca2+ concentration and addition rate in the alginate solution, temperature, intensity and time of agitation during the crosslinking on the homogeneity and stability of the resulting hydrogel.Acknowledgments: The authors are grateful for the financial support provided by the São Paulo Research Foundation (FAPESP), through processes 2022/11767-0, 2022/07967-3, and 2022/03247-6. This research was carried out at the Laboratory of Biomaterials and Biointerfaces (BBLab) of the ITA.References:[1] Cao, H. et al. Sig Transduct Target Ther 6, 426 (2021).

New carbon black-based conductive filament for the additive manufacture of improved electrochemical sensors by fused deposition modeling

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3D printing technology has gained attention in the production of electrochemical (bio)sensors [1]. This expansion is due to the ability to produce devices quickly, and automated. However, there is still much to explore, especially about the main material used in conjunction with 3D printing technology, the conductive filament [1,2]. The production of conductive filaments has gained notoriety, as it allows the production of electrochemical devices. The producing lab-made conductive filaments allows the manufacture of materials with varied compositions and replaces the need to import this product [1,2]. Manufacturing lab-made conductive filaments is an excellent alternative for the production of electrochemical sensors capable. In this work, we report the development of a lab-made filament composed of carbon black and polylactic acid. Optimized filaments containing 28.5% weight of carbon black were obtained and used in the 3D printing of electrochemicalsensors by the fused deposition modeling technique. The manufactured sensors was exploited to detect catechol and hydroquinone in water samples and hydrogen peroxide inmilk. The determination of catechol and hydroquinone was successfully performed by differential pulse voltammetry, showing LOD of 0.02 and 0.2 μmol L^{-1}, respectively, and recovery values ranging from 91.1 to 112% in water. The modification of the sensor withPrussian blue allowed non-enzymatic amperometric detection of hydrogen peroxide, with alinear range between 5.0 and 350 μmol $L^{-1}$and LOD of 1.0 μmol L^{-1}. Thus, the filaments can be applied to build electrochemical sensors capable of being applied for various purposes.

Acknowledgments
FAPESP (2017/21097-3, 2022/06145-0); CAPES (financial code 001; 09/2020, 88887.504861/2020-00); CNPq (301796/2022-0, 380632/2023-3) References

[1] Stefano, J.S., et al., Anal. Chem. V. 94, pp. 6417-6429, 2022.[2] Stefano, J.S., et al., Anal. Chim. Acta., 1191, e339372, 2022.

On the applicability of Energy Density equation for insitu alloying of Ti-Nb-Sn alloys using SLM technique

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The demand for improved load-bearing devices for orthopedic implants has led to the development of novel Ti alloys, particularly metastable β Ti alloys. The choice for Ti alloys is justified by the exceptional corrosion properties, high biocompatibility, and unique mechanical behavior. Recently, the processing of Ti alloys has undergone significant changes due to advancements in additive manufacturing (AM) technology. One of the mostefficient additive manufacturing techniques is selective laser melting (SLM). Usually, SLMtechnology relies on processing costly pre-alloyed powders that have specific compositions,morphologies, and dimensions. The availability of these metallic powders is limited to only a few compositions, making the atomization of aparticular metallic alloy an expensive procedure. An effective approach to surpass restrictions regarding the availability of pre-alloyed powders is the use of In-situ alloying additive manufacturing. In this work, a Ti-30Nb-4Sn (wt.%) alloy was in-situ additively manufactured using different processing parameters. One of the aims of this work was to evaluate how the ranges of scanning speedand laser power, maintaining the same energy density, influence the porosity, homogeneity, and microstructure. The samples were synthesized using Ti, Nb, and Sn powders in an OmniSint-160 SLM machine. The experiments were conducted in an argon atmosphere with scanning speeds varying from 318 mm/s to 1000 mm/s, and laser power between 225 W and 394 W, while maintaining the energy density of 164 J/mm 3 . The compositional and microstructural analyses were performed using optical microscopy, scanning electron microscopy, and energy dispersive spectroscopy. Mechanical properties were evaluated by Vickers hardness tests. The results showed a high variation in the porosity and homogeneity of the samples synthesized with the same energy density, suggesting that the widely used equation does not perform satisfactorily for in-situ alloys.

Physicochemical characterization of AA2017 gas-atomized aluminum powders for its application in LPBF

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Laser Powder Bed Fusion (LPBF) is one of the most used techniques for the construction of metal parts by additive manufacturing. Although the physical and microstructural characteristics of the LPBF parts depend of different factors related to the characteristics of the machine and the processing parameters, the physicochemical characteristics of the raw material used also influence the quality of the built part. In this sense, the characterization and understanding of the physical, microstructural, and morphological characteristics of metal powders destined for LPBF are of great importance. Considering the above, the characterization of gas-atomized powder samples of the AA2017 aluminum alloy was carried out in this work. The particle distribution, moisture, and fluidity of the powder samples were performed by sieving, drying tests, and Hall funnel. The microstructural and morphological characteristics were performed using optical microscopy (OM) and scanning electron microscopy (SEM). Phase characterization was performed by X-ray diffraction (XRD). The results obtained in this work demonstrated the obtaining of powder samples with an optimal particle size distribution and efficiency of the atomization process greater than 90% during atomization, and obtaining 71% of useful powder to be applied within LPBF (between 20μm - 63μm, for this work). The characterization through OM and SEM allowed to observe the formation of predominantly spherical powders, presenting an ideal morphology for its application within LPBF. Nevertheless, the fluidity tests carried out using a Hall funnel did not show a good fluidity of the material. Likewise, the drying stages of the material revealed that the powder samples are prone to capture a large amount of moisture from the environment, which can negatively influence the fluidity of the material, as well as the possible formation of defects in parts built by LPBF due to the formation of oxide layers.

Preparation and characterization of nano-hydroxyapatite paste for additive manufacturing

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Hydroxyapatite, a calcium phosphate-based ceramic found in bone tissue, is one of the most used ceramics to produce feed materials for 3D printing. Due to its excellent biocompatibility and biodegradability properties, it can be used in many areas, such as biomaterials and environmental applications [1]. Hydroxyapatite nanoparticles were synthesized by the sol-gel route using the sonicator technique [2]. Nano-Hydroxyapatite (nanoHAp) pastes were prepared using Poly(Vinyl Alcohol) - PVA and Poly(ethylene glycol) -PEG as organic binders. Firstly, the PVA:PEG, with a proportion of 7:3, was solubilized in distilled water under heating and vigorous stirring. The nanoHAp powder was added to the polymer-based solution at 70, 80 and 90%wt using an Ultra Turrax homogenizer for 10 min. The flowability of the pastes was evaluated by extruding tests [3] using syringes with needles of 0.84 mm in diameter at printing speeds of 5 and 120 mm/min. The microstructure and chemical composition of the nanoHAp powder and paste samples, before and after sintering at 1070 ℃ for 120 min, were evaluated by SEM/EDS and XRD analysis. The results showed that the synthesized nanoHAp powder is composed of agglomerates and particles with an average size of 1.3 ± 0.95 microns and 21.0 ± 4.9 nm, respectively. The paste with 80%wt of nanoHAp exhibited the best flow characteristic for additive manufacturing.

Acknowledgements:
The authors thank to Funding Authority for Studies and Projects (FINEP Process Number 01.022.0282.00 - Ref: 0042/21) and National System of Nanotechnology Laboratories (MCTI/SisNANO/INTCENANO-CNPq Process Number 442604/2019-0) for financial support.

References:

[1] E. ROMANCZUK-RUSZUK, et al., Ceramics, 6, 364-385 (2023).
[2] L. F. CÓTA, et al., Materials Science Forum, 869, 896-901 (2016).
[3] E. FEILDEN, et al., Journal of the European Ceramic Society, 36, 2525-2533 (2016).

Preparing Permanent Magnet Composites using Laser Powder Bed Fusion

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Laser Powder Bed Fusion (L-PBF) is a selective laser sintering technique used in Additive Manufacturing (AM) to create composite materials with a polymer matrix, including permanent magnet materials. Previous research has shown that processing parameters and feedstock characteristics can impact the properties of composite magnets [1, 2]. However, less attention has been given to the interaction between the laser beam and the magnetic particles, and its effect on the microstructure and properties of the fabricated magnet. In this study, Nd-Fe-B powders with varying particle s were mixed with polyamide-12 powder to create feedstocks for L-PBF. The L-PBF process affected the intrinsic coercivity of the powder particles differently, with smaller particles being particularly sensitive to the laser beam's interaction under atmospheric conditions. Based on these findings, an encapsulation method was developed to shield small powder particles from degradation during the L-PBF process. By utilizing this approach, PA composite magnets with particles in the 4 µm range were successfully prepared, with an intrinsic coercivity of 900 kA/m.

[1] Engerroff, J.A.B. et al. Journal of Rare Earths. v. 37, p. 1078 (2019)
[2] FIM, R.G.T. et al. Additive Manufacturing, v. 35, p. 101353 (2020)

Process parameters optimization and characterization of 17-7 precipitation hardness (PH) stainless steel manufactured by L-PBF

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Aerospace components produced through Metal Additive Manufacturing (AM) have a promising future in several industry sectors. Among the AM processes, Laser Powder Bed Fusion (L-PBF) allows savings in finishing processes and enables the creation of complex geometries, in addition to enabling the topological optimization of parts, increasing component efficiency. Precipitation Hardening (PH) Stainless Steels are of great interest to the aerospace industry due to the combination of properties such as high mechanical strength, good ductility and good corrosion resistance [1]. In this work, the optimization of processing parameters of stainless steel 17-7 PH by L-PBF was studied, aiming to achieve high density and low volume of defects. The varied processing parameters were scanning speed (600, 900, 1200 mm/s) and laser power (98, 144, 186, 230 W), while layer thickness, hatching and scanning strategy were kept constant. The samples produced showed relative densities ranging from 60.5% to 99.9%. The highest densification was obtained for the parameter set that has the highest volumetric energy density. Before and after solubilization heat treatment for the sample with the highest relative density, microstructure characterization using techniques of Optical Microscopy (OM), phase quantification by X-Ray Diffraction (XRD) and Vickers microhardness have been performed. To evaluate the corrosion resistance, electrochemical measurements have been performed in 3.5% NaCl solution at 25 °C with a classic three-electrode cell configuration.

Acknowledgments:
To Foundation of Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP)

References:

[1] CAMPBELL Jr, F. Manufacturing Technology for Aerospace Structural Materials. Amsterdam: Elsevier, 2006.

Quick methodology for estimation of X-ray transmission in microtomography for 3D printingmaterials

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X-ray computed microtomography (μCT) is a non-destructive technique used for studying several samples. It allows for the analysis and reconstruction of three-dimensional models based on X-ray projections. However, the ideal condition for X-ray transmission is necessary for the acquisition of each projection image. The Bruker Skyscan 1272 μCT, equipped with a CMOS detector, requires a transmission value between 40 and 15% (for acquisition of ½ of revolution) [1]. In this work, we present a quick methodology to estimate the thickness of 3D-printed samples that exhibit X-ray transmission within this range. Theoretical models that predict transmission decay curves as a function of the sample's thickness were generated using the Lambert-Beer Law. We used a 3D-printed sample with different thicknesses ranging from 1.0 to 5.0 mm, and cubes (edge cubes: 5.0, 10.0, and 20.0 mm), printed in polyamide 12 employing the Selective Laser Sintering technique (SLS) in the HiQ 3D printer. We tested the methodology for all the standard filter configurations present in the Skyscan 1272 μCT. Comparing the theoretical results with the experimental ones, we observed that the models exhibit good reproducibility with recovery values between 90% and 100%. The importance to establish such protocol is the near future offer of this equipment as an open facility, complementing the current ones available at CTI Renato Archer.

Acknowledgments:
We would like to thank FINEP for the EMUTISAÚDE project funding (#01.18.0027.00) and the CNPq/PCI program (process n. 301583/2023-4).

References:
[1] BRUKER. Skyscan 1276 user manual, Cmos Edition. 2021.

Red ceramic waste reuse for application in catalytic reactions

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The ceramic industry is one of the fastest-growing sectors, leading to a considerable accumulation of ceramic waste[1]. Therefore, several ways to reuse these residues in different areas make it a promising material for the industrial area: catalyst, tubular ceramic membranes, etc. Thus, the research aims to study the physicochemical characteristics of catalysts supported on ceramic waste (roof tiles) for potential reuse for printing of structured catalysts by adding manufacturing [2]. After characterization of the wastes, Cu, Ni, and Fe catalysts supported on the roof tiles wastes were synthed by impregnation of nitrate salts of the metals to obtain 10wt.% Cu, Ni, and Fe catalysts, respectively. The support and catalysts were characterized through TGA/DTA, XRF, XRD, N2 physisorption, and H2-TPR. EDX characterization of the tile waste showed the presence of Si, Fe, Al, K, and Ti was observed, with Si being the most abundant in the SiO2 quartz phase, according to XRD analysis. This phase was also found in XRD analysis of the prepared catalysts, besides the characteristic peaks of Cu, Ni, and Fe. TPR profiles of catalysts showed high reducibility at temperatures lower than 600 °C, representing an advantage for industrial applications. The Ni/T C catalyst showed a single consumption of H2 in its TPR profile, with a maximum at 456°C. Two peaks were found in Cu/T, at 344°Cand 454°C, attributed to CuO reduction in two stages. For Fe/T, a two-step reduction wasalso observed, with peaks at 333°C and 440°C. Printing tests, model catalytic and glycerol conversion reactions are under development to study these properties' effect on the catalysts' activity and selectivity.

Acknowledgments: The authors acknowledge the support of: Laboratório de Síntese de Catalisadores, LSCat - UFAL, Laboratório Tecnano - UFALReferences:

[1] Elen M. Oliveira et. al, Journal of Engineering Research and Applications (2015)
[2] O.H. Laguna et. al, Materials & Design, 208, 109927 (2021)

Rheological Properties and Porosity of Cement Pastes Enhanced from Layered Double Hydroxides (LDH) Additions

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The cement industries are among the most important of carbon emissions. As a result, researchers and industry experts are increasingly seeking eco-efficient materials that can improve the sustainability of cement production. One promising approach is using nanostructured additives in cement pastes [1]. Recently, the memory effect of mixed oxide (MO) derived from the calcination of layered double hydroxides (LDH) was used to improve the properties of cementitious materials [2]. This study used LDH based on Mg0,66Al033(OH)2(NO3)0,32·xH2O to prepare MO additives. The impact of specific surface areas (SSA) and the amount of MO additives on the rheological properties of Portland cement pastes was investigated. LDH with different crystallinity was used to obtain MO additives with SSA= 220, 176, 98, and 53 m2 g-1. The results indicate that both parameters (quantity and SSA) influence the character of cementitious materials. The flow tests evidenced a significant shear stress reduction in the paste containing 1 wt% of MO additive. In contrast, the shear stress was increased by the addition of 2 and 3% of MO on the paste. When both parameters are raised, the consolidation time of the pastes is clearly reduced. The additives altered the hydration reactions of the cement paste, which led to a decrease in the pores volume of the cement. The cement additives affected rheology and porosity over time, and this information was used to build a response model. Therefore, the research involving the addition of MO to cement could enhance cement industry sustainability.

Acknowledgements: CAPES, Finep, FAPESP, CNPq, and INCT CEMtec. INCT CEMtec - Advanced Eco-efcient Cement Technologies.

References:

1. K. L. Scrivener, V. M. John and E. M. Gartner, Cem Concr Res, 2018, 114, 2-26.
2. A. A. Almeida, R. M. M. Santos, M. A. Alves Rosa, S. H. Pulcinelli, V. M. John and C. v. Santilli, ACS Appl Nano Mater, 2022, 5, 7896-7907.

Synthesis and characterization of bifunctional CaO-Al_2O_3 mixed oxides for catalytic applications

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Several industrial reactions use catalysts with specific functionalities. Alkaline earth metal oxides are suitable basic catalysts; however, they present disadvantages such as low specific area, low stability, and deactivation[1]. In this work, CaO was modified with Al_2O_3 in molar ratios Al_2O_3/CaO=0.5; 1 and 2 to study the effect of molar ratio on the physicochemical characteristics of solids, emphasizing on acid-base properties, for potential for printing structured catalysts by adding manufacturing[2]. The mixed oxides were synthed by co-precipitation from $Al(NO_3)_3 \cdot 9H_2O$, CaO (produced from egg shells) and NH_4OH. Subsequently, the supports were impregnated with 10wt.%Cu, obtaining Cu/CA0.5, Cu/CA1, and Cu/CA2. The supports and catalysts were characterized by TGA, DRX, EDX, TPD-NH_3, TPD-CO_2, TPR, and N_2 physisorption. XRD diffractograms of the supports showed crystalline phases that differed between the materials, showing that the morphology changed given the molar ratios. TPD-CO_2 profiles showed that the CA2 support mainly presented weak basic sites, while the CA1 and CA0.5 presented a heterogeneous distribution with medium and strong basic sites. TPD-NH_3 profiles showed an opposite trend for acid sites. Thus, it's noted that the difference in molar ratio modified the solid's crystalline structures, altering the acid-base properties. On the other hand, copper catalysts showed CuO reduction peaks with different metal-support interactions. Printing tests, model catalytic and glycerol conversion reactions are under development to study these properties' effect on the catalysts' activity and selectivity.

Acknowledgments:

The authors acknowledge the support of: Laboratório de Síntese de Catalisadores, LSCat -UFAL, Laboratório Tecnano - UFAL and Laboratório de Microscopia Eletrônica de Alta resolução - LaMAR-CAIPE-UFF

References:

[1] Banković-Ilić, I. B. et. al. Renewable Sustainable Energy Rev., 72, 746 (2017)[2] O.H. Laguna et. al, Mater. Des., 208, 109927 (2021)

Synthesis and characterization of Fe_2O_3 mixed oxide bifunctional catalysts

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Metal oxides are widely used in catalytic reactions due to their physicochemical properties. CaO has been used in several reactions that occur through a basic mechanism[1]. Despite its low cost and accessibility, it has disadvantages such as leaching and low specific area. In this work, CaO was modified with Fe_2O_3 in molar ratios Fe_2O_3/CaO=0.5, 1.0 and 2 to study the effect of molar ratio on the physicochemical characteristics of solids, emphasizing on acid-base properties, for potential for printing structured catalysts by adding manufacturing[2]. The solids were obtained by co-precipitation of Fe(NO3)3·9H2O with the proper amounts of CaO, obtaining CF-0.5, CF-1.0 and CF-2.0. After calcination, the solids were impregnated with copper nitrate to obtain 10wt.% Cu catalysts supported on mixed oxides. The solids were characterized by the techniques of XRD, NH_3-TPD and CO_2-TPD. This work achieved good results regarding the formation of mixed crystalline phases identified by XRD, obtaining phases of pure iron oxides, Fe_2O_3, and mixed phases, with different concentrations for the solids. TPD-CO_2 and TPD-NH_3 profiles, showed differences in the distribution of acid and basic sites in the synthed solids. For the TPD-CO_2 analyses, itwas possible to identify the presence of weak and strong basic sites, where with the lowestmolar ratio there was greater CO2 desorption. For the characterization of TPD-NH_3, the opposite was observed regarding the distribution of basic sites, acid sites were more expressive when the Fe_2O_3/CaO molar ratio increased. Printing tests, model catalytic and glycerol conversion reactions are under development to study these properties' effect on thecatalysts' activity and selectivity.

Acknowledgments: The authors acknowledge the support of: Laboratório de Síntese de Catalisadores, LSCat - UFAL, Laboratório Tecnano - UFAL

References:

[1] H. Helmiyati and I. Masriah, AIP Conf. Proc., 2168 (2019)[2]

O.H. Laguna et. al, Mater. Des., 208, 109927 (2021)

Synthesis and characterization of nickel nanorods:effect of pH, precursor, temperature and synthesistime.

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Precious metal-based catalysts are widely used due to their catalytic activity[1]. However, the use of nickel as a catalyst is receiving increasing attention due to its low cost and catalytic activity, similar to precious metals. The present work studied the effect of the reaction parameters in obtaining nickel nanorods. The synthesis was developed by modifying the method proposed by LUO, Y. et al.[2]. Three different precursors $NiC2O4·2H2O$, $NiCO3$ and $Ni(NO3)2·6H2O$, pH between 8 and 14, reaction temperatures between 100 and 180°C and reaction time between 24 and 110 h were used. All syntheses were performed in an autoclave reactor with a teflon coating. The solids were purified by centrifugation at 3000 RPM for 10 min, dried at 80°C for 24 h, and posteriorly characterizedby XRD, SEM, and H2-TPR. The XRD analysis showed that when nitrate and carbonate precursors were used, a crystalline structure of $Ni(OH)2$ was obtained. For the oxalate precursor, it was possible to obtain the crystalline phase of nickel oxalate. Scanning Electron Microscopy showed that nickel rods were only obtained when using Nickel Oxalate, when modifying the synthesis temperature, and different widths when modifying pH. The synthesis time did not show significant differences in the morphology of the rods, but the XRD showed a better crystallinity for the solids synthed in 132 h. The temperature-programmed reduction of the obtained rods showed a reduction peak at 380°C. The best synthesis conditions to obtain rods with uniform diameter distribution and high crystallinity were 180°C, 24 h, and pH=14.

Acknowledgments:
The authors acknowledge the support of: Laboratório de Síntese de Catalisadores, LSCat -UFAL, Laboratório Tecnano - UFAL and Laboratório de Microscopia Eletrônica de Alta resolução - LaMAR-CAIPE-UFF

References:
[1] VAIDYA, P., et al. Chemistry Europe, vol. 2., p. 6563-6576 (2017)
[2] LUO, Y., et al. Trans. Nonferrous Met. SOC. China vol. 16., s96-100 (2006)

The influence of print orientation in the production of porcelain pieces through Direct Ink Writing

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Porcelain has physical characteristics that allow its use in different fields of application, ranging from domestic to industrial. However, the disadvantage is the difficulty of producing complex and detailed geometries. Direct Ink Writing (DIW) is an additive manufacturing technique that uses material extrusion to manufacture 3D structures, allowing the production of customized parts quickly and without material waste [1,2]. In this context, it is important to understand the influence of the layers'' orientation of samples produced using robocasting on their mechanical properties. The products will be developed using the DIW of ceramic materials, being block and cubic specimens. For printing the specimens, three different orientations will be considered, and sintered at 1250ºC for 2 hours. SEM will be used to evaluate the morphological characteristics of the material. The determination of the mechanical characteristics of the material will be done through axial compression and three-point bending tests. In this way, the work seeks to understand the behavior of parts manufactured through DIW regarding the orientation of the layers and the influence of the production of the final properties of the material.

Acknowledgements:
The authors acknowledge the financial support from the Coordenação de Aperfeiçoamentode Pessoal de Nível Superior - Brazil (CAPES) - (contract 88887.663486/2022-00)

References:
[1] ALVAREZ, F. CIFUENTES, A. SERRANO, I. FRANCO, L. FARGAS, G. FENOLLOSA, F. UCEDA, R. LLANES, L. TARDIVAT, C. LLORCA, J. ROA, J.J. Optimization of the sinteringthermal treatment and the ceramic ink used in direct ink writing of α-Al2O3: Characterization and catalytic application. **Journal of the European Ceramic Society**, v. 42 p. 2921-2930, 2022.
[2] LAMNINI, S. ELSAYED, H. LAKHDAR, Y. BAINO, F. SMEACETTO, F. BERNARDO, E. Robocasting of advanced ceramics: ink optimization and protocol to predict the printing parameters - A review. **Heliyon**, v. 8 e.10651, 2022.

The Printability of 316L SS by Laser Powder Bed Fusion and Effects of Laser Power, Velocity, and Scan Strategy on the Density, Microstructure, and Phase Composition

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Laser Powder Bed Fusion (L-PBF) is the metallic Additive Manufacturing (AM) technology with the highest worldwide adoption level by industry. Among the advantages of this technology concerning others, we can mention the selectivity and the possibility of building impossible geometries [1]. Selectivity is directly related to the thermal history and processing parameters that will determine how printable a given powder is and the material properties at the end of manufacture [2]. This investigation aimed to evaluate the effects of main L-PBF parameters on the properties of the 316L stainless steel alloy, one of the most applied materials in the industry: density, hardness, microstructure, and phase composition. A single laser L-PBF machine was used to process cubes with a side of 10 mm using the same gas-atomized powder (with 20 and 53 micrometers in volume, D10 and D90, respectively) and with a layer thickness of 0,060 mm. There were variations of two laser scanning strategies (Continuos and Chess), three power levels (250, 325, and 400 W), three speeds (900, 1100, and 1300 mm/s) and three hatch distances (0.075, 0.105, and 0.135 mm), totalling 54 sets of parameters. The samples were characterized by Image (by Optical Microscopy) and Arquimide´s Densities, Hardness, Microstructure, and Phase Composition (XRD). Significant effects of power, speed, hatch distance and scanning strategy parameters were observed on density and hardness properties. However, no significant effects were observed on the phase compositions of the four set parameters evaluated to compare the two scanning strategies.

References:

[1] Sing, SL.; Yeong, WY. Virtual and Physical Prototyping 15 (2020).
[2] Yadroitsev, I.; Yadroitsava, I.; Plessis, AD.; MacDonald, E. Fundamentals of Laser Powder Bed Fusion of Metals (2021).

Thermomechanical response of 3D Porous Carbon-based Schwarzite Structures

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Schwarzites are crystalline, 3D porous structures with stable negative curvature formed of sp2-hybridized carbon atoms [1]. These structures present topologies with tunable porous size and shape with unusual mechanical properties [2,3]. In this work, we have investigated the thermomechanical behavior under compressive/tensile strains and energy absorption of four P8-family schwarzite structures, through reactive molecular dynamics simulations, using the ReaxFF force field as available in the LAMMPS code. Our results show that under mechanical compression these structures can be reduced up to 80% of their original size before structural failure (fracture) occurs depending on the size of the "graphene patches" in the structures. Our results also show remarkable resilience under mechanical compression and high temperatures (up to 1000 K). We will also discuss recent efforts to synthesize them. REFERENCES: [1] A. L. Mackay and H. Terrones, Nature, v352 762 (1991). [2] L.C. Felix, C. F. Woellner, and D. S. Galvão, Carbon v157, 670, (2020). [3] L. V. Bastos, C. S. Tiwary, D. S. Galvão, and C. F. Woellner - submitted. ACKNOWLEDGEMENTS: The authors thank the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) for financial support through the Programa Institucional de Bolsas de Iniciação Científica para o Ensino Médio - PIBIC- EM.

Ultrasonic tomography to identify the presence of pipesin concrete structures

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Ultrasonic tomography as a non-destructive evaluation method has been increasingly studied in civil engineering since it has the potential of analyzing the interior of concrete structures without damaging them. Special attention has been given to the identification of water and sewer PVC pipes integrated into the structural elements since their presence can interfere with their performance. This work presents a non-destructive method for identifying the presence of PVC pipes in concrete structures using ultrasonic tomography. This technique utilizes ultrasonic waves to generate a sectional image of the concrete structure and detect anomalies. A tomographic reconstruction algorithm based on the Random transform was developed. To validate the proposed method, concrete specimens with known internal flaws were created. PVC pipes with different cross sections were located inside the specimens. Ultrasonic readings were conducted in the specimens, and tomographic images were developed. The accuracy of the images and mesh dependency were also evaluated. Experimental results demonstrate that the proposed technique could accurately identify the location and presence of pipes in the concrete specimens. The presented method could be beneficial for inspecting concrete structures for preventive maintenance and repairing concrete structures with the presence of pipes.

Valorization of Agave Americana fiber-reinforced poly(lactic) acid biocomposites for 3D printing by FDM

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Additive manufacturing (AM) of eco-friendly bio-composites had been growing recently to valorize agricultural waste and obtain green fillers capable of reducing bioplastic costs, without compromising the material processability and performance. In this study, a thermokinetic mixer processed the Polylactic Acid (PLA)/ fibers from Agave Americana (AA) (10% wt.) biocomposites to obtain 3D printing filaments extruded at 180 °C, which were used to print testing samples using Fused deposition modeling (FDM) 3D printer. Filamentswere characterized by Scanning Electron Microscopy (SEM), Thermogravimetry (TGA), Infrared Spectroscopy (FTIR), and Shore Hardness. Filaments revealed homogeneous diameters. The addition of 10% wt. fiber did not significantly influence the filament's diameter and density compared to pure PLA. On the other hand, thermal stability and morphological analysis were influenced by the addition of fiber in the matrix. Also, insertingfibers were uniformly distributed and embedded throughout the wire cross-section compared to the pure PLA due to good interfacial bonding. The composites developed haveshown enhancement in their mechanical properties (shore hardness). Despite the low concentration of natural fiber on the composites investigated in this work, the successfulobtainment of PLA reinforced with biodegradable natural fiber, compromising neither its thermal properties nor its processability and printability, opens the possibility for future work investigation into a composite with larger fiber content.

Wear and corrosion behavior of a boron-modified duplex stainless steel produced by laser powder bedfusion

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Metallic components used in the oil and gas industry are often subjected to severe corrosion and wear degradation. Most of these parts are made of stainless steels, which present high corrosion resistance but relative low wear resistance [1]. Several studies have been developing strategies to improve the wear resistance of stainless steels, such as the addition of high boron content (0.3-2 wt.%), which is responsible for the improvement of the wear resistance due to the formation of hard particles of borides [2, 3]. The production of duplex stainless steels by laser powder bed fusion (LPBF) has earned considerable interest owning to the capability to produce in a noticeably short time near-net-shape components with complex geometries. For the first time, a duplex stainless steel modified with 0.3 and 0.6 wt.% of boron was produced by LPBF and its microstructure (Scanning electron microscospy/EDX), corrosion (Cyclic potentiodynamic polarization, and electrochemical impedance spectroscopy), hardness (Vickers) and wear (reciprocating pin-on-plate test) behavior were evaluated and compared to a commercial duplex stainless steel. The 3D-printed samples present high density, a refined microstructure and the hardness increases by increasing the boron content. Acknowledgments:Fundação de Amparo à Pesquisa do Estado de São Paulo/FAPESP (scholarship n° 19/05885-7 and 2021/10735-4), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior/CAPES (Finance code 001). The Austrian aviation program ‘ ‘TAKEOFF” (PILOT, grant number 852796, 2018) and BMK - The Austrian Ministry for Climate Action, Environment, Energy, Mobility, Innovation and Technology.

References:[1] A. Bahadori, Wiley, New York, 2014.[2] J. Soyama, G. Zepon, T.P. Lopes, L. Beraldo, C.S. Kiminami, W.J. Botta, C. Bolfarini, J. Mat. Resear., v. 31(19), p. 2987-2993, 2016.[3] J. Soyama, T.P. Lopes, G. Zepon, C.S. Kiminami, W.J. Botta, C. Bolfarini, Met. Mat. Inter., v. 25, p. 456-464, 2019.

R-Organic Electronics, Photonics and Bioelectronics: Fundamentals, Applications and Emerging Technologies

Capacitive field-effect sensors for biosensoricapplications

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Among the multitude of concepts and different types of chemical sensors and biosensors discussed in literature, the strategy to integrate chemical or biological recognition elements together with semiconductor-type field-effect devices is one of the most attractive approaches. In this context, typical examples are represented by the capacitive EIS (electrolyte-insulator-semiconductor) sensor, the LAPS (light-addressable potentiometric sensor) or the ISFET (ion-sensitive field-effect transistor).These different kinds of devices are currently being the basic structural element in a new generation of chemical and biological microsensors, fabricated by means of silicon planar technology. Moreover, they provide a lot of potential advantages over conventional approaches such as the small size and weight, the fast response time, the possibility of an on-chip integration of sensor arrays, the high robustness, the possibility of low-cost fabrication, etc. At the same time, their possible field of application reaches from medicine, biotechnology, process control and environmental monitoring through food and drug industries up to defense and security requirements.This paper gives an overview on recent examples of silicon-based field-effect (bio-)chemical sensors developed at the Institute of Nano- and Biotechnologies. The sensors have been dealing with different receptor molecules such as enzymes, polyelectrolytes or DNA molecules, respectively, as well as device concepts up to biomolecular logic gates. Additionally, various immobilization strategies will be presented and discussed.

Femtosecond-laser processing of diamond for photonic applications

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The integration of devices suited for applications in photonics and quantum information technologies demands the development of platforms that combine, in the same structure, resonators, waveguides, and materials with desirable features. Diamond has generated interest as a material to be incorporated in such devices because defects in its crystalline structure may present phenomena of interest in the quantum information field as well as photonics. One of the most promising solid-state optically active spin systems is the nitrogen-vacancy (NV) center in diamond, which is usually produced by irradiating ultrapure synthetic diamond with beams of electrons or nitrogen ions. NV centers can be optically initialized in a well-defined quantum state, presenting long coherence times at room temperature and allowing creating protocols to manipulate its spin-state using a combination of optical and magnetic resonance techniques. NV centers can also be used as quantum sensors to detect physical parameters, such as temperature, magnetic and electric fields. Also, NV centers in diamonds have emerged as a promising platform for integrated photonic circuits, combining their nonlinear optical properties with excellent thermal properties. The controlled production/placement of NV centers in optical structures is of foremost importance, because the ability to engineer such devices can be widely used in quantum optics, photonics, magnetometry, thermometry, etc. In this direction, this work demonstrates the use of fs-laser pulses not only to generate active and spatially localized NV centers in diamond, but also to fabricate polymeric optical microcavities incorporated with nanodiamonts containing NV centers, that support whispering gallery modes (WGM). We also determined the damage threshold fluence for the diamond surface at distinct wavelengths and the influence of the number of pulses on the center generation.

Meniscus-Guided Coating of Organic Semiconductorsfor Applications in Field-Effect Transistors

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Solution processable organic semiconductors are promising for large area, lightweight, and flexible field-effect transistors. Control over their film microstructure and molecular organization is crucial for the charge carrier transport in transistors. Meniscus-guided coating (MGC) covers techniques like zone-casting, dip-coating, and solution shearing, and is an efficient approach to solution process semiconductors into highly ordered thin films. Over the recent years, various MGC methods have been developed to precisely tune the crystallization of semiconducting small molecules and polymers to further improve their charge carrier transport. MGC is classified into two deposition regimes, evaporation and Landau-Levich, with a transition region as mixed regime according to the relation between coating speed and the film thickness. During processing, the evaporation rate and coating speed play a significant role on the crystal growth of the molecules. Mixed solvents also permit tuning their solubility, nucleation rate and crystallization. The wettability and surface energy of the substrate also influence the nucleation and crystal growth of the organic semiconductors during meniscus-guided coating. This presentation discusses the crystallization and film growth mechanism of organic semiconductors during meniscus-guided coating that is essential for the thin film deposition for field-effect transistors. An understanding on the alignment mechanism and fundamental principles of the fluid mechanics for the crystal growth has been developed. Relations between meniscus shape, fluid mechanical process and semiconductor crystallization during solution processing are provided. Homogeneous mono- and multilayers based on conjugated polymers and small molecules have been fabricated for transistor applications through a careful control of the processing conditions.

This work was supported by the National Science Centre, Poland, through the grant UMO-2019/33/B/ST3/1550.

Solid state solvation: matrix effects in TAD-OLED

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Dyes of interest for advanced in OLED and OPV applications are extremely sensitive to the dielectric properties of the local environment. Solid state solvation plays a major role in this context and must be understood and fully exploited in the concurrent optimization of the dye inside the host matrix towards optimized devices. Here we present a joint experimental and theoretical work that sheds light on the dielectric response and relaxation of organic matrices of interest for TADF-OLED application.

Spin recombination statistics limits the internal quantum efficiency of organic light-emitting diodes (OLED) to a disappointing 25%. TADF beats the efficiency limit in purely organic systems, towards low-cost and easy to recycle devices. In TADF-OLED, non-emissive triplet states, generated upon charge injection, are converted into emissive singlets via reverse intersystem crossing (RISC).The dielectric response of the matrix and its rigidity largely and non-trivially affect the TADF photophysics. This intricate problem is faced focusing on a prototypical TADF dye, described by an essential state model, parametrized ab initio and validated against spectroscopic data in solution [1]. On this basis, we propose an original approach to address RISC rates fully accounting for the non-adiabatic coupling between electrons and conformational degrees of freedom [2] and finally to address the delicate issue of the TADF photophysics in non-polar and polar matrices [3].

Acknowledgements:

This project received funding from the European Union Horizon 2020 Research and Innovation Programme under Grant Agreement No. 812872 (TADFlife) PNRR MUR project ECS_00000033_ECOSISTER

References:

[1] R. Dhali, et al., Phys. Chem. Chem. Phys., 2021, 23, 378.

[2] D. K. A. Phan Huu, et al, J. Mater. Chem. C, 2022 10, 4620.

[3] D. K. A. Phan Huu, et al., J. Amer. Chem. Soc., 2022, 144, 15211.

Analytical Approach for the Analysis of Multivariate Impedance Data

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Functionalized sensor arrays are widely used in electronic tongue/nose systems to detect and sense chemical compounds. These arrays generate multivariate complex impedance data, which are analyzed based on the global selectivity principle to qualitatively discriminate substances and their corresponding concentrations. However, commonly used analytic methods such as principal component analysis (PCA) and interactive document map (IDMAP) are non-quantitative, making it impossible to develop calibratable devices. To address this issue, we propose a novel approach for analyzing multivariate impedance data from sensor arrays used in electronic tongue devices. Our method allows for the representation of data in a quantitative 2-D space, where devices produced using similar fabrication parameters exhibit similar values when exposed to the same compound at the same concentration. To demonstrate the effectiveness of this approach, we conducted an extensive set of experiments involving the impedance (capacitance) vs. frequency spectra from 8 different types of electrodes functionalized with organic semiconducting films and 5 types of analytes at different concentrations. We also tested triplicated devices to confirm the reproducibility of the electrical impedance response. Our approach successfully discriminated between different analytes using unspecific electrodes for detection, with good reproducibility of the response. Additionally, the concentration dependence of the electrode array response allowed us to determine the detection limit for each compound and each detection calibration curve. This method can be easily extended to smart sensing applications, including biosensors, that require reliable and quantitative analysis. Acknowledgement Fapesp process 2019/08019-9.

Changes observed on performance and parameters extracted from electrolyte-gated transistors dependenton gate voltage

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Electrolyte-gated transistors (EGTs) have in their structure a dielectric electrolytic layer. Many of these electrolytes depict biocompatibility bringing the versatility of applying them as e.g.: neuromorphic devices, basic building blocks in bioelectronics or biosensors. Their electrical characterization depends on appropriate models to correctly extract the parameters. In this work, we depicted a sequence of measurements performed on EGTs operating as EGOFET and OECT from two distinguished EGTs, whose electrolyte is composed by: (i) Ion gel (P(VDF-HFP) and [EMIM][TFSI]) analyzed under different gate voltage range and different sweeping time during the scan measurement (ii) Hydrated gelatin used as a platform to add E. coli bacteria in an EGT-sensor, whose signal was quantified by the transistor' s extracted parameters. The experimental results obtained from these different devices are analyzed using two different models. Despite the major differences between devices and modes of operation, the OFET model is still largely used in literature to extract EGOFET parameters and often showing to be a misappropriated model for this transistor class. An possible alternative is the LS model which brings information about some extracted parameters dependence with $V_G$for EGOFETs and OECTs, not considered in the OFET model. This model also indicates some microscopic phenomena as, e.g. most likely region for ion diffusion into the transistor' s channel. The LS model brings a new point of view for the extraction of some parameters, a situation of extreme importance for the correct quantification/interpretation of experimental data, here quantified on two applications: a solid ion gel-EGT and an EGT-sensor.

Acknowledgments:
The authors thank to FAPESP and CAPES.

References:
[1] G. Horowitz, Springer Berlin Heidelberg, (2010) pp. 113-153.
[2] Luginieski, M. et al. Adv. Theory Simul. 2200852 (2023).

Conformation of polyethyleneimine (PEI) interlayers used in organic electronics probed by SFG vibrational spectroscopy

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Polyethyleneimine (PEI) is a versatile cationic polyelectrolyte that is a surface modifier widely used as an interlayer in organic electronics. Ultrathin layers of PEI on electrodeslead to the formation of interfacial dipoles that result in a reduced work function (WF) andtherefore could improve charge injection and device efficiency. There are two possible explanations for such WF changes: (i) the preferential ordering of polar groups at the interface and (ii) the interfacial charge transfer from the conductor to the organic semiconductor. However, a detailed experimental evaluation of the nature of this dipole layer has not yet been reported. Here we used sum-frequency generation (SFG) vibrationalspectroscopy - a non-linear optical technique intrinsically specific to interfaces and sensitiveto the orientation of polar groups - to investigate the conformational organization of PEIultrathin layers. We have studied films fabricated by dip-coating from aqueous solution andby spin-coating from isopropanol solution onto gold films, and for comparison, we also probed the organization of PEI deposited on glass, which is a non-conductive material. Theinfluence of the dip-coating solution pH on the PEI interfacial organization was also investigated. The obtained SFG vibrational spectra indicate the existence of conformationalordering at all interfaces, but only after several days after PEI deposition. This is likely aresult of a slow drying of the deposited layer. The spectra are more intense (more orienteddipoles) for dip-coated films that were fabricated from solutions with pH ~ 5 for glass andpH ~ 2 for gold, while spin-coated films tend to have less conformational order. These results are consistent with the mechanism for WF change based on molecular dipole orientation, rather than interfacial charge transfer.

Acknowledgments

We acknowledge funding by FAPESP (2020/13917-3, 2018/15670-5, 2014/50869-6), CNPq (315772/2020-4) and CAPES (88887.607212/2021-00).

Designing emitters bases on Cu(I) complexes with thermally activated delayed fluorescence and phosphorescence

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Speeding up the emission lifetime in luminescent copper(I) complexes has been highly challenging due to the copper atom''s relatively low spin-orbit coupling constant compared to heavier metals such as Iridium. We recently reported a [Cu(NN)(PP)]+ complex with 1,10-phenanthroline-derived containing the selenodiazole moiety, which showed thermally activated delayed fluorescence. Interestingly, this complex did not show the expected "heavy atom effect, enhancing the spin-orbit coupling matrix elements. Nevertheless, theaddition of the heavier chalcogens contributed to reducing the photoluminescence lifetimeto roughly 800 ns, which is the lowest reported so far for such [Cu(NN)(PP)]+ materials [1].This also enhanced the performance of solution processed OLEDs [2]. Moreover, the copper-iodide (Cu-I) moiety''s effect on the phosphorescence decay pathway was investigated. Temperature-dependent photophysical studies revealed a combined thermally activated delayed fluorescence and phosphorescence emission, with a phosphorescence decay rate inthe order of 104 s-1, which is higher than usual for copper(I) complexes. Density functionaltheory calculations indicated very high spin-orbit coupling matrix elements between the low-lying states of these Cu-I complexes comparable to the extensively applied coordinationcomplexes based on heavier metals. The Cu-I demonstrated to be a versatile moiety to speedup the phosphorescence decay pathway in about one order of magnitude [3]. Blue emittersbased on copper(I) complexes were also synthed, combining high emission efficiency, shortlifetime, and easy synthesis.

Acknowledgements:

The authors thank to CNPq, CAPES, FAPESC, INCT/INEO and OCTA.

References:

[1] Giliandro Farias et al., J. Mater. Chem. C, 2020, 8, 14595.

[2] Cristian A. M. Salla et al., Eur. J. Inorg. Chem. 2021, 3412-3418.

[3] Giliandro Farias et al., Dalton Trans., 2022, 51, 1008

Direct Laser Writing Fabrication and Thermo-Optic Characterization of Polymeric Microresonators

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Polymeric photonic devices have gained great scientific and technological interest due to their ease of processing and functionalization. Here, we focus on fabricating high-quality polymeric Whispering Gallery Mode (WGM) resonators using the two-photon polymerization (2PP) technique. WGM resonators are of great interest in optical sensing and filtering applications due to their high-Q factors and compact size. We used a Ti:Sapphire laser centered at 780 nm with a 50 nm bandwidth, 86 MHz repetition rate, and 100 fs pulses to fabricate WGM resonators doped with the azoaromatic compound DR13. The resulting microstructures were of high quality, with good structural integrity and low roughness on the walls. Taper fibers were produced for coupling light into these structures, and then the coupling to doped WGM microresonators was made. The spectra of the resonators showed high-Q factors. The effects of doping the structures with the organic DR13 compound for absorbing irradiation were also verified. When the doped structures were irradiated using a 532 nm laser, a significant modal shift was observed. Additional studies are being performed to determine which heating factor is dominant: (I) changing of the material density, and consequently its refraction index, or (II) expansion of the resonator, or even both. A direct analysis by changing the temperature of the substrate is being performed to establish a relationship between laser power and effective heating of the resonators. Also, we are performing thermal and thermomechanical analysis, respectively, to determine the transition temperatures and the polymer thermal expansion coefficient. In summary, we demonstrate successful fabrication of high-quality polymeric WGM resonators using 2PP and investigate the influence of thermal and expansion effects on the modal distribution of the structures. Our work has potential applications in basic science and in optical data transmission and microbiology sensors technologies.

Electronic Transport in Organic Solar Cells: J-VAnalytical Models and Electric Transient Measurements

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The three current types of third photovoltaic generation - organic, perovskites, and quantum dots - are promising technologies for the production of clean and renewable energy in the near future. This presentation is devotes to bulk-heterojunction organic solar cells (BHJ- OSCs), in which the active layer is thin film of a nanostructured morphology, formed by electron donor and electron acceptor molecules. The enormous area of contact between thedonor and acceptor phases considerably increases the generation of carriers available forelectrical conduction, but, at the same time, it also enhances the probability ofrecombination between positive and negative photocarriers. Carrier recombination is one ofthe main factors limiting the efficiency of BHJ-OSCs. The classic stationary J-V (voltage- current) characterization provides us with the necessary parameters to calculate cell efficiency (short-circuit current Jsc, open-circuit voltage Voc, and fill factor FF), but offerslittle information about the mechanisms of generation and transport of carriers. Therefore,the elaboration of models and the search for alternative and complementary experimentsare necessary. In this sense, we present two J-V analytical models, one considering first-order recombination kinetics [1], and another second-order kinetics [2]. A new characterization technique will also be discussed: transient measurements [3]. To analyze the results, we use classic BHJ-type cells: ITO/PEDOT:PSS/P3HT-Th:PC61BM/Ca/Al and ITO/PEDOT:PSS/PTB7-Th:PC71BM/Ca/Al. Acknowledgements: This work was carried out in the National Institute for Science and Technology on Organic Electronics, which is supported by FAPESP and CNPq. [1] D. J. Coutinho et al., Solar Energy Materials Solar Cells 143, 503-509 (2015) [2]D. R. B. Amorim et al., Phys, Rev. Appl. 14, 03446 (2020) [3] M. R. P. da Cunha et al., Solar Energy Materials Solar Cells 231 (2021) 111313

Enhancement of NLO response of organic chromophores through solvent effects: Theoretical Modeling using a multiscale methodology

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Theoretical Modeling of new materials with higher nonlinear optical (NLO) activity is an important goal in photonics and optoelectronics. In particular, a challenge in Photonics and Optoelectronics is to take advantage of specific environmental forces to enhance the nonlinear optical response of π-organic materials. Under this statement, we present a systematic study using multiscale methods [1, 2], combining classical simulations and Quantum mechanical calculations to describe the substitutional effect of some chemical groups and the solvent effects on the linear and NLO properties of a series of organic compounds that should have promising uses as organic light-emitting diodes. The results reveal that the environment can also be used to tune a chromophore's optical response, leading to an enhancement of up to 10 times the NLO response of the molecule. Such studies can help in the selection of potential candidates for NLO applications, providing a valuable contribution to the development of more efficient and sustainable optoelectronic materials.

Acknowledgements:
This work has been supported by the following Brazilian research agencies: CNPq, CAPES, FAPEAL.

References:

[1] V. Manzoni et al. Optical Materials 94, 152-159 (2019).
[2] V. Manzoni et al. Journal of Molecular Liquids 335, 115996 (2021).

Exploring Homotruxene derivatives as potential liquid crystal anisotropic emitters with delayed fluorescence

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The combination of blue delayed fluorescence (DF) and mesomorphic characteristics, such as presented by liquid crystals (LC), is highly interesting in the development of new organic light emitting diodes (OLEDs), because a LC can be aligned and this results in a luminescence external quantum efficiency (EQE) enhancement, along with a simplification of the device [1,2]. In this perspective, this work aims to incorporate the properties of LCs in homotruxene (HTX), while preserving the distinctive DF features of this highly promising polycyclic aromatic hydrocarbon [3]. It was observed that some compounds synthed so far present a behavior that resembles that of an LC, but that the data are so far not completely understood because of the low phase transition temperatures. However, X-ray diffraction technique at different temperatures are being carried out to better investigate the mesomorphic behavior. Time-resolved emission spectroscopic analyses (TRS) at 90 K and at room temperature show that in solution some of these materials exhibit DF (418nm or 3.0 eV) and phosphorescence (600 nm or 2.1 eV) at room temperature. The results suggest that although the characteristics necessary for a good DF are opposite to those necessary for mesomorphism, the structural proposals presented here, derived from HTX, are on the way to the simultaneous stabilization of both. In a second phase of this project, new compounds containing perfluorinated chains are being synthed. The idea is that this will both hinder non-radiative relaxation paths and favor nanosegregation of the different molecular sections, which is necessary for good DF and LCs properties, respectively.

Acknowledgements:

CNRS/Université de Bordeaux, PPGQ/UFSC, GPM3, INEO, FAPESC, CAPES/Cofecub and CNPq.

References:

[1] Lee, J.-H. et al. J. Mater. Chem. C 7 (2019) 5874-5888.

[2] Eccher, J. et al. ACS Appl. Mater. Interfaces 7 (2015) 16374-16381.

[3] Salla, C. A. M. et al. Angew. Chemie Int. Ed. 58 (2019) 6982-6986.

Exploring hybrid nanostructured films as an interfacefor energy storage and sensing applications

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The integration of distinct (nano)materials is attractive for the study and development of new devices with improved properties. In this sense, carbon and metal oxide-based materials have been investigated due to their potential for several technological applications, including energy storage and (bio)sensing [1-3]. Here we present the fabrication of Layer-by-Layer (LbL) and Langmuir-Blodgett (LB) films consisting of different arrangements of graphene oxide with metal oxide nanoparticles incorporated in electrodes to create suitable interfaces capable of acting in supercapacitors and (bio)sensors devices. The use of different metal oxide nanostructures (e.g. MnO_2 and ZnO) and how they were prepared and embedded into graphene sheets and combined with polyelectrolytes to form LbL films or phospholipids to form LB films will be presented, in addition to details of film fabrication and characterization methods. The supercapacitor approaches will be focused on the modification of conductive electrodes, while the sensor studies will be related to the preparation of sensing units in field effect devices. Moreover, the benefits of the arrangement of graphene oxide and metallic oxides in LbL or LB fashion films will be presented, as well as their potential properties to be explored as supercapacitors or (bio)sensors devices.

Acknowledgements:
FAPEMIG (Grants APQ-01464-18 and BPD-00880-22) and CNPq (Grant 437785/2018-1).

References:

[1] D. A. Oliveira, L. H. S. Gasparotto, J. R. Siqueira Jr., An. Acad. Bras.Cienc., 91, No. e20181343 (2021)
[2] O. N. Oliveira, J. R. Siqueira Jr., F. N. Crespilho, L. Caseli, Appl. Mater. Interfaces, 6, 14745–14766 (2014)
[3] S. Shleev, E. González-Arribas, M. Falk, Curr. Opin. Electrochem., 5, 226-233 (2017)

Exploring the Potential of Spray-Coated Graphene Oxide Memristors for Organic Electronics Applications

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Memristors are a rapidly emerging technology for information storage and processing devices. Unlike other electronic components such as resistors, capacitors, and inductors, memristors can remember the amount of charge that has previously flowed through them. This means that they can be used for information storage and processing, making them useful for a wide range of applications, from computer memory to artificial intelligence. Memristors have the potential to be smaller and more energy-efficient than traditional electronic components. This is because they can be made using nanoscale materials, and they don't require a constant supply of power to maintain their state. In this context, Graphene oxide is a promising material for developing organic memristors due to its unique electrical and mechanical properties. Our research investigated Carbonyl-rich graphene oxide (CGO) based memristors using a simple and scalable spray coating method. This method offers advantages over other fabrication methods as low cost, the ability to producelarge-area films, and is compatible with various substrates, including flexible substrates, which can enable the development of flexible and wearable memristor devices. Our memristors exhibited excellent stability and reliability, with repeatable resistance-switchingbehavior over hundreds of cycles at a low operation potential of 1 volt and a high differencecurrent range between a high-resistance state and a low-resistance state. We areinvestigating the mechanism behind resistance switching by analyzing the structural and chemical changes in the CGO film during device operation. The hypothesis is that the migration of the oxygen functional groups in the CGO film causes resistance switching. Ourpreliminary results demonstrate the feasibility of using a simple spray coating method tofabricate CGO memristors for various applications in organic electronics and show significant potential as next-generation electronic devices.

Fabrication and comparison of electrochemical sensors based on polymeric and carbon materials

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This work presents the fabrication and characterization of a flexible electrochemical sensor composed of polymer poly(butylene adipate-co-terephthalate) (PBAT) and Carbon Nitrite Dots (CNDs) or graphite. The materials were characterized by Cyclic Voltammetry (CV), FTIR and contact angle. FTIR analysis of the PBAT/CNDs composite allowed verifying that the insertion of the CNDs in the polymeric matrix modified the spectrum when compared to neat PBAT, since the composite spectrum presents characteristic bands of the CNDs, at 3200 cm^{-1}, attributed to the stretching vibrations of the OH groups and another at 3420 cm^{-1} corresponding to the absorption of the aromatic compound and C-H stretching vibration in aromatic rings. Based on the comparison of the spectra, a modification was observed in the spectrum of the composite based on PBAT and CNDs, indicating the presence of CNDs in the polymer matrix [1]. The measured values of the contact angle for PBAT, exhibited values of the 89.5° ± 1.8°, and the PABT/CNDs electrode was 60.1° ± 0.2°. CV and EIS were usedto compare the electrochemical behavior of the sensor produced using PBAT/graphite (g-PBAT), and the PBAT/CNDs electrodes with and without the application of UVB light. g-PBAT electrode showed higher electrochemical activity compared to the electrode with PBAT/CNDs. However, it was taken into account the amount of material used for the development of electrodes was, since PBAT/CNDs used 10% of CNDs in proportion m/m compared with g-PBAT electrodes the amount of graphite used to 200% in proportion m/m, So the PBAT/CNDs electrode allowed the development of a sensor that uses little material, with low cost and accessible materials. These sensors simultaneously detected emerging pollutants such as cadmium and copper, including real tap water samples, in the concentration range of 0.1 to 1.0mM.

Acknowledgments:
Fapesp (2014/50869-6, 2020/12659-0.

References:

[1] P. Y. Liyanage et al., Bioconjug. Chem., 30, 111-123 (2019).

First-Principles study on the absorption characteristicsof charge-transfer states in α-sexithiophene:fullerene systems

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The interconversion dynamics between charge-transfer-state charges (CTCs) and separated charges (SCs) is still an unresolved issue in the field of organic photovoltaics and has therefore remained highly debated in the literature [1]. Although, ultrafast techniques, such as transient absorption spectroscopy (TAS), bring invaluable insights [2,3], the attribution of the obtained absorption characteristics to the charged species often require knowledge about the photophysical properties of the system and its components. Such information canbe obtained from time-dependent density functional theory (TD-DFT). In this conference contribution, a case study on α-sexithiophene:fullerene (α6T:C60) systems will be presented,for which TAS measurements revealed two charge species with distinct absorption characteristics and different dynamics. Modelling the different charge transfer configurations that occur in system with different morphological structures, TD-DFT simulations identified the species as CTCs and SCs. It is found that the spectral differences arise from broken symmetry in the charge transfer state that turns forbidden transitions into allowed ones. Based on this assignment, a kinetic model could be formulated allowing the characterization of the charge generation, separation, and recombination mechanisms.

Acknowledgements:
The funding through the Fundação de Amparo à Pesquisa do Estado de São Paulo (2018/15670-5), the Swiss National Science Foundation (200020_184819), the Deutsche Forschungsgemeinschaft (EXC2089) .

References:

[1]Shivhare, R. et al. Adv. Mater 2022, 34 (22), 2101784.
[2] Moore, G. J. et al. J. Phys.Chem. Lett. 2020, 11 (14), 5610-5617.
[3] Zhong, Y. et al. Nat. Com. 2020, 11 (1), 833.

Insertion of Carbon Nanotubes in Langmuir-Blodgett films of phospholipids and galactose oxidase for bioelectronics

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Inserting carbon nanotubes in Langmuir-Blodgett (LB) films of enzymes confined in lipids can improve their catalytic performance, which is interesting for bioelectronics since robust biosensors can be assembled. In this work, we incorporated the enzyme galactose oxidase and carbon nanotubes from the aqueous subphase in fatty acid Langmuir monolayers and transferred them to solid supports as LB films to essay the catalytic activity towards the catalyzes of the oxidation of D-galactose. Surface pressure-area isotherms, surface potential-area isotherms, polarization-modulated infrared reflection-absorption spectroscopy, and Brewster angle microscopy characterized the floating monolayers. The adsorption of the enzyme at the air-water interface expanded the lipid monolayer, altering the film compressibility at high surface pressures. PM-IRRAS spectra showed amide bands, revealing the structuring of the enzyme into helices and sheets. The monolayers were transferred to solid supports as LB films and characterized with fluorescence spectroscopy and quartz crystal microbalance. The catalytic activities of the films were measured and compared to the homogenous medium. Viability as sensors was tested with voltammetry and impedance measurements. The enzyme accommodated in the LB films preserved the catalytic activity, and carbon nanotubes helped conserve the catalytic properties for moreextended periods of storage. The method presented in this work allows for enhanced catalytic activity and can help explain why specific film architectures exhibit improved performance.

Acknowledgments:
FAPESP and CNPQ

Layer-by-layer film containing reduced graphene oxide and manganese dioxide nanocomposite for supercapacitor electrodes

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Seeking for new materials combinations and approaches are essential to achieve new nanocomposites with enhanced performance. Carbon materials and metal oxides have been used in energy storage as the their ideal combination can lead to devices with improved power and energy densities [1-3]. Here we present the fabrication of a layer-by-layer (LbL) film containing manganese dioxide (MnO2) nanostructures embedded into reduced graphene oxide (rGO) sheets and arranged with poly(allylamine-hydrochloride) (PAH) for supercapacitor application. Scanning electron microscopy images confirm the incorporation of MnO2 nanostructures into the rGO layers, and cyclic voltammetry and galvanostaticcharge-discharge measurements reveal the electrocapacitive features of the films. Nanostructured PAH/rGO-MnO2 LbL films containing 20 bilayers lead to a supercapacitor with high areal capacitance of ca. 112 mF/cm2 at 1 mV/s and 460 F/g at 1 A/g, in addition to a high capacitive retention performance of 99% over 10,000 cycles and a high charge-discharge time of ca. 600 s. These findings show that the presence of manganese dioxide improved the capacitance, which was twice higher than a blank system of PAH/rGO. Such properties demonstrate, therefore, that the PAH/rGO-MnO2 LbL film is a promising architecture to be further explored in energy storage nanostructured systems.

Mapping the electric field within polymeric transistorsby sum-frequency generation microscopy

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Organic transistors have the potential to enable novel types of devices and applications that were previously impossible with traditional inorganic materials. In order to fully exploit the potential of these devices, it is crucial to investigate the physical and chemical events that occur within them. In this context, understanding the distribution of the electric field across the dielectric and semiconducting layers of an operating organic field-effect transistor (OFET) could provide valuable insights, particularly for comparing with theoretical models of these devices. In our work, we employ sum-frequency generation (SFG) vibrational spectroscopy [1] to probe the C=O stretch in the polymeric dielectric (PMMA). SFG is a non-linear optical effect that is very sensitive to breaking of inversion symmetry, such as the molecular alignment induced by an intense electric field. In addition, this technique offers the advantage of preserving the integrity of the device and avoiding any modification of the probed charge distribution. In this contribution, we will present SFG images obtained from OFET channels that utilize P3HT and PMMA as the organic semiconductor and organic dielectric, respectively. They are effectively mapping the spatial charge distribution within the OFET channel under operation. If time allows, we also will provide preliminary electrical and SFG microscopy measurements obtained from ambipolar OFETS based on PDBT-co-TT. With this study, we hope to contribute for a better understanding of the physics of OFETs, with important implications for improving their performance.

Acknowledgements:
The authors acknowledge funding by CNPq (315772/2020-4 and 442239/2017-3), CAPES (88887.506483/2020-00), FAPESP (2018/02819-0) and the INCT on Organic Electronics - INEO (CNPq/FAPESP: 2014/50869-6).

References:

[1] A. G. LAMBERT; P. B. DAVIE; D. J. NEIVANDT, Appl. Spectrosc. Rev., 40, 103-145 (2005)

Nonlinear optical characterization of dipolarImidazo[4,5-b]pyridine molecules

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In recent years, organic materials have attracted significant attention due to their potential for applications in a range of optoelectronic devices, such as optical switches, modulators, and frequency converters [1,2]. In addition, a number of molecular design techniques are being used to enhance the linear and nonlinear optical (NLO) properties in such materials, achieved through structural modifications and chemical design [2]. The imidazo[4,5-b]pyridine (Impy) molecules, primarily recognized for their biological and biochemical activities, have emerged as a promising nonlinear material for multiphoton absorption processes (MPA) due to their planar structure, high electronic density, and ease of synthesis [1,2]. Specifically, Multiphoton absorption has practical applications in various fields, such as 3D optical data storage, photodynamic therapy, microfabrication, and others. For this reason, this study is oriented to investigate the linear and NLO properties of six impy derivates and to evaluate how the introduction of electron-accepting and donating groups modifies its photophysical properties. The optical characterization, including the two-photon absorption (2PA) process, was performed using the Z-scan technique and other spectroscopic techniques. As a main result, an increase in most photophysical parameters was observed in molecules with a dipolar structure D-π-A in relation to molecules with A- π -A structures. Similarly, D- π -A molecules showed higher values for the 2PA cross-section, indicating less influence of electron acceptor groups on the 2PA properties.

References:

[1] Pelosi, A. G. et al. Influence of electron-withdrawing groups in two-photon absorption of imidazopyridines derivatives. Dye. Pigment. 198, (2022).

[2] L. H. Zucolotto Cocca et al., "Two-Photon Emissive Dyes Based on Push-Pull Purines Derivatives: Toward the Development of New Photoluminescence Bioprobes," Journal of Physical Chemistry C, vol. 124, no. 23, pp. 12617-1

Nonlinear refraction spectrum of resins used in direct laser writing

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In recent decades, the field of photonics has seen exponential growth, reaching applications ranging from optical communication to medical devices. This success lies in the continuous exploration of new ways to manipulate light and the study of optical properties of materials that can be integrated to further photonics applications. This progress highlights the importance of the optical characterization of materials with technological potential. In this regard, materials that exhibit third-order nonlinear phenomena attract much attention. Their understanding has given essential steps in advancing new technologies, such as high-resolution microscopy, all-optical switching, and direct laser writing, to name a few. Among third-order effects, Kerr nonlinearities are one of the most studied effects, being described by the intensity-dependent refractive index $n = n_0 + n_2 I$. For this reason, the spectrally-resolved characterization of the nonlinear refractive index n_2 allows new materials to be identified as candidates for different photonic applications. In this work, we present the n_2 spectra of polymers that have been used in direct laser writing technologies [1]. In particular, we used resins obtained by photopolymerization and studied how different monomer compositions affect the n_2 by using the nonlinear ellipse rotation technique from the visible and near-infrared range [2]. We compared the n_2 thus obtained with reported values for fused sílica, the most widespread material used as a nonlinear reference, and we found values significantly higher for the polymers, demonstrating the potential of these materials.

Acknowledgments:
We thank the financial support from FAPESP, CNPq and the National Institute of Science and Technology in Photonics (INFo).

References:
[1] Adriano J. G. Otuka et al., 3, 024003 (2021).
[2] M. L. Miguez et al., 22, 25530-25538 (2014).

OLEDs for lighting: is simplifying the structure the best way?

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Lighting technology is changing rapidly towards an enhanced area of research looking for the best solutions to get highly efficient, long-life and architectural appellative products. Solid State Lighting technologies based on Organic Light Emitting Diodes (OLEDs) became the focus of huge scientific research in recent years due to their unique properties. In particular, the white-OLED can be fabricated, using the unique properties of organic semiconductor carrier dynamics, in a simple device with only an active layer. In a similar way, signage, employing large area OLEDs in different color emission, are also in focus. However, several drawbacks, particularly focusing on emission efficiency and uniformity as well as thermal stability, should be overcome.

With the new generation of organic emitters based on the Thermally Activated Delayed Fluorescence (TADF) process, a new framework has been opened. With a theoretical internal efficiency of 100% due to full triplet harvesting, these organic materials appear to be the best candidates for large-area OLEDs. This work focuses on the large area (16cm2) OLEDs based on TADF emitters. The materials used are the 2- [4-(diphenylamino) phenyl] - 10, 10- dioxide-9H - thioxanthen-9- one (TXO-TPA, red emitter), the (2,5 -Bis(4-(10H -phenoxazin-10-yl)phenyl)-1,3,4-oxadiazole) (DHPZ-2BI ,green emitter) and 10,10'-(4,4'-(4-phenyl-4H -1,2,4-triazole-3,5-diyl)bis(4,1-phenylene))bis(10H -phenoxazine) (2PX-TAZ, blue emitter). The results show good efficiencies, up to 18% of external quantum efficiency (EQE), in only three layers device. An explanation about the physical process is provided, considering both charge carrier injection/transport limits and involving the host: guest molecular interaction, as one the fundamental key to the success. Device simplicity versus efficiency, points to the viability of simple structures, opening new frameworks. Further pathways to improve large-area OLEDs efficiency are discussed.

Organic mixed ionic electronic conductor based onwater-soluble melanin/PEDOT:PSS blend to bioelectronics and energy storage applications

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Organic mixed ionic-electronic conductors (OMIECs) have gained attention due to their capability to efficiently transport and couple both ionic and electronic charges, specially to bioelectronics and energy storage devices applications [1]. Poly (3,4-ethylenedioxythiophene): poly (styrenesulfonte) (PEDOT:PSS) is the benchmark OMIEC, which shows a high electronic mobility but a lower volumetric capacitance in comparison with other organic materials [1]. Eumelanin is a biopolymer that possesses a variety of carboxylic acid moieties [2] that are capable to uptake cationic species in aqueous media and favoring the ionic-electronic coupling in the polymer [2]. Different mass ratios of synthetic melanin (Mel) and PEDOT:PSS have been characterized by a variety of techniques, such as atomic force microscopy (AFM), Fourier Transform Infrared (FTIR) and UV-Vis spectroscopies, electrochemical impedance spectroscopy (EIS), among others. Current-voltage and EIS measurements show that the electronic conductivity of the PEDOT:PSS is preserved (87 ± 24 S $cm^{-1}$to 2 ± 1 S cm^{-1}) while the volumetric capacitance of the films reaches values of 1.5 x 103 F cm^{-3}, showing a clear improvement of the ion-electron coupling in such samples. Finally, we exploited the potential of PEDOT:PSS/Mel blends in an organic electrochemical transistor (OECT) and a supercapacitor (SC). The OECTs exhibit state-of-the-art transconductance (10.8 ± 2.9 mS) while SC showed specific capacitance of 8 F g^{-1}, and 0.62 ± 0.01 Wh $kg^{-1}$and 190.3 ± 5.7 W $kg^{-1}$of specific energy and power, respectively. We believe that the blend of melanin with PEDOT:PSS can be a powerful strategy towardsnew development of OMIECs and related applications, for example, in sustainable iontronicsand bioelectronics.

Acknowledgements:
FAPESP (2022/00410-3) and LNNano-CNPEM.

References:

[1] Bryan D. Paulsen, et al., Nat. Mat.,19, 13-26 (2020).
[2] João V. Paulin and Carlos F. O. Graeff, J. Mater. Chem. C, 9 (2021), 14514).

Reservoir computing: Using conjugated polymers for constructing physical reservoirs

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Reservoir computing is a new concept that emerged in the early 2000s, which employs the nonlinear dynamics of physical systems, or reservoirs, as a neural network. Unlike traditional neural networks, which require pre-training of all elements, a non-classical reservoir operates without training. The reservoir takes advantage of the nonlinear dynamics of its elements to transmit information, and only the outlet layer of the reservoir is trained for the desired regression or classification [1]. In this research, physical reservoirs were created using electropolymerized conducting polymer microfibers based on EDOT. The authors found that EDOT microfibers were easily grown using a sinusoidal voltage with amplitudes between 2 and 10 V and frequencies ranging from 25 to 200 Hz. Physical reservoirs were then built by placing a monomeric solution in contact with several gold electrodes and applying a sinusoidal voltage to enable polymeric fibers to grow and connect to the electrodes. The physical reservoirs made using EDOT were evaluated for their varied dynamical responses, fading memory, and nonlinear transformations. Additionally, a single fiber of PEDOT was grown on patterned golden substrates, enabling the authors to use the fiber as an Organic Electrochemical Transistor (OECT) [2] and extract information about the transfer and output curves. These classical characterization curves can be utilized to measure performance in OECTs, providing information about the non-linear behaviors coming from a single fiber, which is the elementary persona in a physical reservoir built with polymer fibers.

References:
[1] Nakajima, Kohei Hauser, Helmut Li, Tao Pfeifer, Rolf. (2015). Information processing via physical soft body. Scientific reports. 5. 10487. 10.1038/srep10487.
[2] Rivnay, J., Inal, S., Salleo, A. et al. Organic electrochemical transistors. Nat Rev Mater 3, 17086 (2018). https://doi.org/10.1038/natrevmats.2017.86

Screened optimally tuned range separated hybridfunctionals for solvated molecular systems

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Since the introduction of the optimally tuned range separated hybrid (OT-RSH) functional,[1] density functional theory (DFT) and time-depedent DFT (TD-DFT) have predicted ground and excited state properties with remarkable accuracy. It relies on splitting the Coulomb operator into short and long range terms, with the short range being represented by a local or semilocal approximation and the long range by the exact Fock exchange (imposing the correct asymptotic on the xc-potential, 1/R). The range separating parameter (ω) is chosen such ionization energies are as closest as possible to frontier orbitals eigenvalues (an exact property). However, the representation of chemical environment has some caveats. A non-periodic molecular system can be represented by a multimolecular system and by optimally tuning such large system one can correctly describe its ''''solvated'''' properties. Though quite accurate, this later strategy is computationally expensive. An alternative is to combine the functional optimization with a continuum solvation model. In the standard implementation of these methods the point charges representing electrons and ions are both free to react to the solute''s charge density, thus, only partial ionization energies can be calculated, leading to an exceedingly low ω. Refaely-Abramson et al. have demonstrated that by employing an asymptotic constrain of the OT-RSH functional to 1/ε the Coulomb attenuation is correctly represented in periodic systems.[2] In this presentation, we overview these incompatibilities and present a screened OT-RSH functional that comply with the concept of optimally tuning in a chemical environment. Its performance is attested by contrasting experimental ionization energies and optical gap with calculated ones for molecular systems of interest in organic electronics.

Acknowledgements:
FAPESP and CNPq

References:

[1] T. Stein et al., J. Am. Chem. Soc., 131,8, 2818 (2009)
[2] S. Refaely-Abramson et al., Phys. Rev. B 88, 081204

Study and optical characterization of the poly (3-methythiophene) electrochemically synthetized

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The semiconductor polymer Poly (3-methylthiophene) (P3MT) is highly efficient, inexpensive and electrically conductive, which increases the interest in researching it, as it is within the sustainable consumption standards that scientists and companies seek to develop. The objective of this work is to carry out the electrochemical synthesis of P3MT, to study the optical and structural properties via ultraviolet-visible absorption (UV-Vis), photoluminescence (PL) and excitation photoluminescence (PLE) spectroscopy, to analyse the emission, absorption, energy and bandgap bands. As a result, the UV-Vis spectra were obtained, which made it possible to get the average energy gap of the P3MT, presenting the π-π* transition band and a bipolaron band. Thus, it was possible to characterize and quantifythe energy transfer mechanisms of photoexcited carriers. Furthermore, the cyclic voltammetry data made it possible to calculate the P3MT electronic bandgap. The main results found were: (1) the energy gap value for samples synthesised with $Et_4NBF_4$is 2,18 eV-575 nm and for the electrochemically reduced one 2,10 eV-590 nm; (2) the π-π* transition band of the electrochemically reduced sample compared to the oxidised sample shows a slight shift towards lower energie (3) through the normalised PL spectrum, the deconvolution is obtained, considering two contribution channels. Therefore, it can be concluded that the technique used for the electrochemical synthesis of poly(3-methyl thiophene) is generally simple and easy to understand and that films with the more significant formation of P3MT are more electronegative, which increases the possibility of being applied in electronic devices, such as optical sensors, but further studies are needed to be applied in electronic devices, such as photovoltaic cells.

Acknowledgements:
CNPq, CAPES, UFMT, and FAPEMAT.

Study of the influence of the dopant in the PAni thin film on the energy transfer of the PAni/P3OT interface

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Conjugated polymers have optical and electrical properties of interest for application in organic electronic devices such as OLEDs and OPVs. Polyaniline (PAni) can be synthed as a thin film and has several oxidation states, in which the emeraldine salt state is the most conductive. The regioregular Poly(3-octyl-thiophene) (P3OT) has photoluminescent and electroluminescent properties and a high solubility and can be quickly deposited in the form of a thin film through various techniques. To form the polymeric interface of PAni and P3OT thin films, PAni was first deposited by cyclic voltammetry using a 0.1 M concentration of two different dopants: hydrochloric acid (HCl) and sulfuric acid (H2SO4). PAni has deposited on a fluorine-doped tin oxide (FTO) substrate. The regioregular P3OT was dilutedin chlorobenzene in a 1/0,1 ratio by mass and deposited on the PAni films by spin-coating. Samples of pure PAni and P3OT films were deposited on FTO to compare with the interfaces. UV-Vis absorption measurements were made and the spectra show a maximum absorbance of 520 nm for the P3OT film and 350 nm for the PAni films. The UV-vis spectrum of PAni-HCl does not show the intense polaronic band at 800 nm, which indicates fewer defects. The absorbance spectrum of the PAni/P3OT interface showed an overlap of the PAni and P3OT spectra. Photoluminescence (PL), atomic force microscopy (AFM), emission ellipsometry (EE), and electrochemical impedance spectroscopy (EIS) measurements will be made. Through photophysical, electrochemical, and morphological measurements, it is possible to analyze the influence of the dopant on the deposition of PAni on the PAni/P3OT interface, to evaluate the transfer of energy between the polymeric chains. Finally, the interest of this study is to analyze the polymer/polymer interaction of the PAni/P3OT interface.

Acknowledgements:
CAPES, CNPq, and FAPEMAT.

Synthesis and Characterization of New Potential TADF Copolymers for the Development of OLED Devices

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Organic Light Emitting Diodes (OLED) are widely studied for their advantages. One of the main challenges is the formation of emitters with pure color and high efficiency [1]. The investigation of new polymeric emitters is of great interest in the scientific community, as is the case of thermally activated delayed fluorescence (TADF) emitters [2]. This project focuses on synthesizing new TADF copolymers with two different acceptor units, diphenylsulfone, and anthraquinone, to obtain color emitters, blue and red, respectively. A 2-step synthetic route was followed: first, the addition of the acceptor to the 2,5-dibromocarbazole nitrogen by SN2 (M1, M2). Then the reactive bromine side chain was changed to the boronic acid pinacol ester unit (M3, M4). The copolymers were produced by the Suzuki-Miyaura reaction between M3 or M4, (9-(9-heptadecanyl)-9H-carbazole-2,7-boronic acid bis(pinacol)ester) and a spacer (1,4-dibromobenzene or 1,4-dibromo-2,3,5,6-tetrafluorobenzene). The proportion of units varied between 20:30:50 and 10:40:50, respectively. 1H- and 13C-NMR spectra of the monomers exhibited predictable characteristic peaks, and the polymers showed broad peaks as expected. TGA measurements showed high thermal stability (>400 °C). Furthermore, the UV-Vis spectroscopy results showed absorbance in the blue region. The anthraquinone-containing polymers exhibited a redshift compared to the others. The polymer band gap was around 3.0 eV suggesting a blue emission, and slightly lower for the red polymers. Furthermore, the polymers showed the expected color emission under 365 nm irradiation. The emission mechanism of the polymers will be studied, and later, these materials will be applied as an emission layer in new OLEDs.

Acknowledgments:
The authors thank the funding agencies CNPq, Capes, and FAPERJ for their financial support.

References:

[1]S.J. Zou and Y. Shen, et al., Mater Chem Front. 4., 788-820 (2020).
[2]X. Yin and Y. He, et al., Front Chem. 8., 1-23 (2020).

Synthesis of PEDOT:PSS Conductive Inks: Do Byproducts of the Chemical Route Influence the FinalProperties of the Ink?

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PEDOT:PSS, a type of conducting polymer widely utilized in organic electronics, has gained significant attention in various applications, including electrodes, supercapacitors, and sensors, owing to its distinctive properties. Despite extensive research on PEDOT:PSS, onlya limited number of publications have examined the relationship between the properties of conductive inks and the parameters of their synthesis, highlighting a topic that remains largely unexplored. Specifically, there is insufficient knowledge about the influence of chemical byproducts on the final properties of PEDOT:PSS. In a previous study, it was demonstrated that the variation of oxidant agent (OA) during the synthesis of PEDOT:PSS has an impact on the charge density of the final polymer films.[1] This effect was attributed to the reduced rigidity of PSS−(aq) chains, brought about by the screening of electrostatic interactions of sulfonate groups (SO3−) with OA ions. Consequently, the ability to couple charged PEDOT+ with the PSS− counterpart is diminished. However, it remains unclear whether the counterion of PSS− also affects the collective dynamics of charged chains in solution and its implications for the electrical properties of PEDOT:PSS.

This study presents the results of Dynamic Light Scattering (DLS) measurements of PSS solutions with different counterions, including the poly-acid (PSS−-H+) and the poly-salt (PSS−-Na+) forms. Additionally, the electrical characterization of PEDOT:PSS/PSSNa filmswas conducted using the four-point probe method. The DLS measurements revealed that PSSNa chains exhibit lower electrostatic rigidity compared to PSSH chains. Furthermore, itwas observed that the same addition of OA to the solution causes greater contraction in chains of the poly-salt than those of the poly-acid, further impeding the coupling of PEDOT+and PSS−. This observation aligns with the findings from the four-point probe, which indicate that PEDOT:PSS ink synthetized with PSSNa show conductivity values that are 10times lower than those using PSSH.

Acknowledgements

The authors acknowledge funding by CAPES finance code 001 and INCT on Organic Electronics (CNPq/FAPESP: 2014/50869-6).

References

[1] H. F. P. Barbosa, G. D. G. Higuita, F. Günther, G. C. Faria, Advanced Electronic Materials 2022, 8, 2100864.

The case study of Nematic – Smectic C transition investigation in Isoxazoles and Isoxazolines 3,5-diarylsubstituted Liquid Crystalline Esters.

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Due to their large exocyclic bond angle 5-membered heterocyclic rings are at the edge between rod-like and bent-shaped liquid crystals (LCs). Polar properties that emerge in the macroscopic structure in this type of materials of the LCs are dependent on this geometrical parameter. Such structural features tends to show a distinct nematic (N) phase, being called bent-core nematics (BCNs), characterized for a local biaxiality by smectic clusters (cybotactics) formation into the nematic phase (Ncyb) or twist-bend nematic (NTB) mesophase.[1][2] In this work we have synthesized a new set of 3,5-diarylisoxazoline/isoxazole esters in which the overall shape of the structures are at the boundaries of rod-shaped and bent-shaped LCs. The liquid crystals behavior was investigated and the data were compared with the diarysubstituted oxazadiazoles.[3] The liquid-crystalline behavior was characterized by POM, DSC and XRD studies. All molecules exhibit LC phases, except one derived from isoxazoline ring. Nematic mesophase as detected with a helicoidal growing during the transition to the SmC phase for most of the esters investigated in this study.

Acknowledgements:
We are grateful to MCTI/CNPq N° 309661/2020-0 853, CNPq/Ed. Universal 403.134/2021-8), Fapergs/EditalPqG 07/854 2021 21/2551-0002121-7 for the financial support and A. R. P. thanks to and PIBIC/CNPq/UFRGS for his fellowship.

References:
[1] F. Vita, Liq. Cryst., 43, 2254 (2016).
[2] O. Francescangeli, E. T. Samulski, Soft Matter, 6, 2413 (2010); L. D. Lopes, Crystal Growth Design, 2023; https://doi.org/10.1021/acs.cgd.2c01101
[3] G. Shanker et al., ChemPhysChem, 15, 1323 (2014).; L. Chakraborty et al., J. Mater. Chem. C, 1, 1562 (2013); S. Singh et al., New J. Chem., 40, 6834 (2016).

Thin films of Polyaniline-based nanocomposites with CeO2 and WO3 metal oxides applied to the impedimetric and capacitive transducer stages in chemical sensors

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Although extensive research has been conducted on polymeric nanocomposites for sensor applications, the use of PANI-based nanocomposites in impedimetric and capacitive electrochemical sensors remains largely unexplored. The objective of this study is to investigate the relationship between the composition of nanocomposites and the sensitivity and linearity of electrochemical impedimetric and capacitive pH sensors. PANI/CeO2 and PANI/WO3 thin films were produced through spin coating, characterized and subsequently used in electrochemical impedance spectroscopy (EIS) and capacitance spectroscopy (ECS) platforms. The results showed an optimized performance for the devices depending on the composition of the sensing thin film. Specifically, PANI/CeO2 composite exhibited a 213% increased ECS sensitivity when compared with pristine CeO2 and an increase of 64% in ECS linearity of PANI/WO3 in relation to the polymeric sensitivity. Despite not as optimized as ECS, the EIS method also resulted in an incremented sensitivity for the composites: 20% for PANI/CeO2 and 37% for PANI/WO3 when compared with the sensitivity of the metal oxides. Furthermore, this study investigated the structure-property relation of the considered materials using standard techniques such as scanning electron microscopy (SEM), contact profilometry and absorption UV-Vis spectroscopy. The findings of this work offer promising prospects for future studies on the application of PANI-based materials as chemical sensors.

Acknowledgements:
Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (Capes), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Fundação de Apoio à Pesquisa andLabMic-UFG.

References:
[1] Rossignatti, B.C.; Vieira, A.P.; Barbosa, M.S.; Abegão, L.M.G.; Mello, H.J.N.P.D, Polymers, 15, 578 (2023).
[2] Poddar, A.K.; Patel, S.S.; Patel H.D., Polym. Adv. Technol. 32(12), 4616-4641 (2021).
[3] Sen, T.; Mishra, S.; Shimpi, N.G., RSC Adv., 6, 42196-42222 (2016).

Ultrathin Surface-Supported Metal-Organic Framework Heterojunctions for Novel Hybrid Electronics Based on Rolled-up Nanomembranes

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Surface-Supported Metal-Organic Frameworks (SURMOFs) are crystalline materials based on metallic clusters and organic linkers layer-by-layer grown on functionalized surfaces.[1] These hybrid structures have the intrinsic high porosity and mechanical flexibility of Metal-Organic Frameworks (MOFs).[1] This unique set of co-related properties opens exciting alternatives to create novel hybrid electronics. However, the typical high porosity can result in glassy interfaces and layer disorder during device integration whether conventional evaporation methods are employed. Therefore, one of the ongoing challenges for these structures relies on the elaboration of appropriate methods of integration. In this direction, nanomembrane-origami technology can be a promising tool. This technology can be designed to create rolled-up nanomembranes, providing self-adjustable top contacts on ultra-thin films without physical damage.[2] In this work, we demonstrate the integration of ultrathin HKUST-1 SURMOF heterojunctions using rolled-up nanomembranes as the top electrode. This concept allowed us to access unprecedented physical effects, namely, ambipolar resistive switching and negative differential resistance.[2,3] Employing the same approach, we integrated these structures into a 3D ultracompact capacitor structure to determine their intrinsic dielectric properties.[4] In addition, the experimental results obtained are interpreted with insights from density functional theory (DFT) calculations, ab initio molecular dynamics simulations, and finite-element calculations. These findings show promising perspectives for SURMOF thin-film structures toward novel hybrid electronics.

Acknowledgments:
FAPESP (2017/25553-3), CAPES, and CNPQ.

References:
[1] Shekhah, O. et al. J. Am. Chem. Soc. 129, 15118-15119 (2007).
[2] Albano, L.G.S. et al. Nano Lett. 20, 1080-1088 (2020).
[3] Albano, L.G.S. et al. Small 17, 2101475 (2021).
[4] Silva, R.M.L. et al. Adv. Electron. Mater. 8, 2200175 (2022).

Unraveling Fundamentals on Organic Electrochemical Transistors Through Thermodynamics Principles

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In recent years, Organic Electrochemical based devices (OEDs) have been the key focus of many developments, mainly in the fields of neuromorphic and bioelectronics [1]. Despite the successful implementation of OEDs, the fundamental processes that regulate their operations and sensing capabilities, specifically those related to ion-to-electron transduction, remain unclear [2]. Consequently, there is a lack of robust models to explain the steady-state and transient characteristics of OEDs and electrochemical biosensors [3]. In this study, we bring new highlights to a thermodynamic-based model that quantitatively describes OEDs'' operation, with a special focus on the Organic Electrochemical Transistor (OECT). Here, we present alternative interpretations to drain current traditional models, based on thermodynamic principles. Beyond that, we conducted a systematic study on the influence of the electrolyte composition on P3HT-based OECT, and analyzed the results in light of the proposed thermodynamic model. By analyzing a large amount of experimental data with distinct electrolytes, we generated guidelines for material design and device development, targeting highly sensitive electrochemical biosensors and devices.

Acknowledgments:

We acknowledge CAPES, INEO, EESC, and IFSC-USP for the financial support.

References

[1] J. Rivnay, S. Inal, A. Salleo, R. M. Owens, M. Berggren, and G. G. Malliaras, Nature Reviews Materials, 3, 2018.

[2] Paudel, P. R., Tropp, J., Kaphle, V., Azoulay, J. D., Lüssem, B. Journal of Materials Chemistry C, 9(31), 2021.

[3] R. Colucci, H. F. de P. Barbosa, F. Günther, P. Cavassin, and G. C. Faria, Flexible and Printed Electronics, 5, 2020.

Virucidal action of photogenerated phenothiazineradical cation impregnated in cotton fabric and polypropylene face mask.

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The advent of the pandemic has led to growing interest in self-disinfecting materials, mainly because other variants and similar viruses may appear in the future[1]. In the present study, for the first time, it was demonstrated that natural and synthetic fabrics could gain virucidal properties after the impregnation of a stable free radical. Cotton and polypropylene fabrics were modified with the photochemically generated 10H-phenothiazine (PHT) radical cation adsorbed to promote virus elimination. The impregnation of PHT radical cation was evidenced by the UV-visible spectrum showing the fingerprint band at 530 nm[2], FT-Raman spectral changes, and the appearance of radical cation EPR signal. The EPR signal of PHT-treated cotton fabric persisted and intensified after the fabric washing and re-irradiation. Colorimetric test evidenced the persistence of radical cation in PHT-treated cotton fabric even after 2 months in the dark. Viral inactivation assays were performed with the cotton fabric and the polypropylene face mask treated with PHT. Results of qPCR showed that cotton fabric impregnated with PHT radical cation presented a two hundred twenty-four-fold reduction in the viral genome compared to the original cotton fabric. Face masks treated with PHT reduces genome copies by ten times. Oxidative attack of PHT radical cation on virus envelope was evidenced by the quenching of EPR signal of fabrics exposed to a model of virus envelope and by cryo-TEM (cryo-Transmission Electron Microscopy) images showing virus destruction. One advantage of the pro-oxidant attack is the non-specificity that allows efficiency against different viruses and bacteria.

Acknowledgements:

Supported by FAPESP 2017/02317-2 and CNPq 313111/2021-9 (ILN) and CAPES 88887.513142/2020-00

References:

[1] WHO. Tracking SARS-CoV-2 Variants. Who. 2021, p https://www.who.int/en/activities/tracking-SARS-Co.

[2] SANTOS, H. et al. Dyes and Pigments, v. 177, 108314 (2020b).

A drift-diffusion model based on fundamental material parameters for organic electrochemical transistor devices: steady-state and transient description

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Among all the different devices of organic electronics, organic electrochemical transistors (OECTs) are particularly interesting as they have emerged as potential transducers in applications that require the conversion of ion fluxes to electronic current [1], For the rational optimization and understanding of the fundamentals of OECTs and OECT-based applications, however, it is essential to have theoretical models capable to predict and recreate the experimental data. In our work, we formulate a model that describes the ion flux from electrolyte into the semiconducting layer based on drift and diffusion by making use of intrinsic material parameters. While our model exhibits an analytical solution for steady-state conditions, the differential equation to describe the transient behavior requiresa numerical treatment. In doing so, our approach goes beyond other drift-diffusion models[2], which exhibit limitations as they making use of boundary conditions that enable analytical solutions. In particular, our approach allows simulating the doping level along the source-drain channel, a non-uniform electric field and non-constant diffusion coefficients as consequence of the material differences between electrolyte and semiconductor. In this contribution, we present simulated steady-state properties such as the drain current as a function of the gate voltage and compare our results to other models from literature. We found qualitative good agreement to trends of the recently published thermodynamic model [3], although the modeling approach is fundamentally different. Furthermore, the impact of the model parameters on the transient responses will be presented and analyzed.

Acknowledgements:
Funding by FAPESP (2018/15670-5), INCT (CNPq/FAPESP: 2014/50869-6) and CAPES finance code 001 is acknowledged.

References:

[1] Flex. Print. Electron. 5 (2020), 013001.
[2] Sci. Rep. 4 (2014), 1.
[3] Nat. Comm. 13 (2022), 4514.

Al/MEH-PPV/ITO organic memory device: characterization and ambient effect

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Devices were prepared using an ITO bottom electrode (BE), MEH-PPV film as the semiconductor layer, and a 100 nm thick aluminum layer was thermally evaporated as the top electrode (TE). Electrical measurements, current vs. voltage (I-V), and spectral impedance were performed with the device under vacuum and ambient conditions. The electroforming process occurred under a vacuum and was monitored using both techniques. I x V measurements show diode-like behavior when the device is pristine. At the end of this electroforming, two conductive states are observed: the HRS (High Resistance State) and LRS (Low Resistance State). The observation of these two conductive states was considered memory behavior. In this situation, the device switch between both states using precise voltages on direct and reverse polarizations, classifying the device as bipolar [1]. In contrast to the pristine state, the LRS and HRS states are characterized by ohmic transport. Impedance measurements were also intercalated into I-V sweeps to study the frequency response of the device. Before memory behavior is observed, the device exhibits effects that are not purely capacitive. Once memory behavior is observed, LRS and HRS states have well-defined responses fitted by an equivalent circuit model, which is an association of a resistor in series with a resistor and a constant phase element (CPE). A CPE element is characteristic of different transport processes with different time scales, consistent with the development of conducting filaments [1]. After characterization under vacuum, samples were exposed to ambient conditions and characterized again. The impedance of the LRS and HRS states approach each other and relax slowly to the pristine state in approximately 2 h. To see LRS and HRS states again a new electroforming process under vacuum is necessary.

Aknowledgments: INEO, CNPq, CAPES, FAPEPI, UFPI, and FINEP.

References:

[1] Yeol Yun et. al. Applied Physics Letters, vol. 104, no 2, 2014.

Alternative biocarbon counter-electrode for optical electronic device

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Facing the environmental challenges of energy crisis and global warming, the use of carbon materials has emerged in the development of renewable energy due to their excellent conduct properties, high physicochemical stability and promising application as counter-electrodes substituting expensive noble metals, such as Au or Ag. In addition, materials obtained from biomass sources have increased due to their wide availability, low-cost processability, adjustable band gap that ensure efficient charge transfer between the interfaces of a photovoltaic device, compared to other materials based on biomass [1,2]. Biocarbon counter-electrode enable the use of recyclable waste of organic matter to reduce the environmental impact. In this work, biocarbon were produced through the carbonization of biomass from sources of sugarcane bagasse and were introduced into the carbon paste to replace the traditional carbon black derived from oil residues. The carbon paste is prepared by ball-milling a mixture of carbon black, graphite powder and $ZrO2$ powder [3]. Carbon black plays a crucial role on the photovoltaic work function, which affects the charge extraction and eventually influences the overall device efficiency. With the resulting carbon paste, films were devised and analyzed for crystallinity and biocarbon morphology, characterized by X-ray diffraction and scanning electron microscopy. Were investigate the work function, sheet resistance and Interfacial connection as well. The use of metallic electrodes is a major obstacle to be faced for the upscaling of solar cells, through that perspective the biocarbon material is a promising substitute for the expensive Au and Ag CE for low cost solar cells.

Acknowledgements:

To the CNPq and CETENE for the resources and infrastructure for the research.

References:

[1] Kohlrausch et al., Energy Technology vol. 11, page 14 (2022)
[2] C.Liu et al., Solar RRL vol. 5, (2021)
[3] G. Liao, Z. Liu, T. Shi, Z. Tang and B. Sun, Nanoscale, page 6 (2015)

Asymetric Donor-Acceptor molecule exhibiting an efficient Thermally Activated Delayed Fluorescence: Photophysical study

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Pure fluorescent emitters employ only singlet excited states for eletroluminescence, limiting its efficiency up to 25%, determined by spin statistics. Thermally Activated Delayed Fluorescence (TADF) mechanism allows the harvest of non-emissive triplet states into emissive singlet states via intersystem crossing process. Reached when decreasing the ∆stat KbT energy level that requires a spatial separation of the High Occupied Molecular Orbital (HOMO) and the Low Unoccupied Molecular Orbital (LUMO) in the molecule. Charge transfer state, in Donor-Acceptor molecules, is a way to promote this spatial separation. Here, we present the optical spectroscopy investigation and concept proof, of a pure organic small molecule, based on a phenoxazine (donor) and dibenzothiophene-S,S-dioxide (acceptor). Photoluminescence may vary from an efficient TADF in function of polarity environment.[1,2] The experimental works comprises the time resolved measurements, performed in solution, as well as in solid state, inert matrix, in fucntion of temperature, e.g. time resolved spectra. The basics photophysics were also investigated, in order to fully explore its potential.

Ackonwledgements:
This work was supported by: CAPES, CNPQ, PGf-UNIFEI, UNIFEI and FAPEMIG project APQ00052-21.

References:

[1]-Ward. J. S. et al. The interplay of thermally activated delayed fluorescence (TADF) and room temperature organic phosphorescence in sterically-constrained donor-acceptor charge-transfer molecules. Chem. Commun. 52, 3-6 (2016).
[2]- Nobuyasu R. S. et. al. Rational design of TADF ploymers using a Donor-Acceptor monomer with enchanced TADF Efficiency induced by the energy alignment of charge transfer and local triplet excited sates. Adv. Opt. Materials (2016).

Bioimpedance-based Biosensors

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The latest innovations in biosensors and emerging applications have been used to deliver easy-to-use diagnostic systems for healthcare. Today's leading biotechnology companies are developing novel semi-synthetic ligands, aptamers, peptides, and conducting polymers to produce functional systems that can deliver precise molecular information for early diagnosis of diseases. Biosensors are powerful tools for environmental, human and animal health monitoring, especially when they are used with bioimpedance spectroscopy for in situ measurements to determine contaminants in fluidic samples. The objective is to present the Bioimpedance Analysis (BIA) using biosensors as miniaturized and disposable detection devices for specific chemical (or set of chemicals), biomolecules [1], adulterants and microorganism analysis in different fields concerning health point-of-care, food quality [2], food safety and pollution. For that, this work aims to gather original articles and reviews showing research advances, fabrication, innovative applications, new challenges and future perspectives of BIA-based (Bio)sensors in important areas as biomedical engineering, health [3], IoT, agri-food and environmental. BIA has been successfully used for cancer diagnosis and biomaterial characterizations due to its safety, low cost, effectiveness, portability and applicability. The results have been showed that miniaturization of such a device depends on the application, portability, electrode geometry, number of electrodes and working frequency bandwidth. It can be concluded that a biosensor can be applicable to detect the biochemical and biological samples if we fulfill the requirements of simplicity, robustness and sensitivity.

Characterization of thin films of a columnar liquid crystal based on perylene diimide deposited by spincoating and blade coating techniques

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Liquid crystals (LC) are materials which present intermediate phases between the crystalline solid and the isotropic liquid and are promising organic semiconductors for presenting self-organization and the possibility of molecular alignment on electrodes, providing greater mobility of charge carriers[1]. In this work, we present the characterization of a brand-new thermotropic discotic LC (designated BPD-11) derived from benzoperylene diimide with a hexagonal columnar mesophase at room temperature and investigate its properties in thin films produced by spin coating and blade coating techniques. The mesomorphic characterization of the material was performed by polarizing optical microscopy (POM), differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA) and x-ray diffraction (XRD). The thin films were prepared from a solution using chloroform as solvent. The morphological, structural and optical properties of the films were performed and the results indicated that these films are very attractive for optoelectronic devices applications such as in OLEDs and solar cells.

Acknowledgements:

CNPq, CAPES-COFECUB, INCT/INEO, FAPESC, OCTA and UFSC.

References:

[1] Gupta, R. K., Manjuladevi, V., Karthik, C. Choudhary, K. Thin films of discotic liquid crystals and their applications. Liq. Cryst. 43, 2079-2091 (2016).

CLASSICAL MOLECULAR MECHANICS APPROACH TO THE INTERMOLECULAR INTERACTION POTENTIAL BETWEEN P3HT:PC₆₁BM COMPLEX

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Charge and energy transport in low-mobility materials represent a challenge for organic photovoltaic devices (OPVs), which makes understanding the potential for interaction between the donor and acceptor species indispensable. In this work, we carried out a study of the intermolecular interaction potential between the complex formed by a regioregular poly(3-hexylthiophene) oligomer (oligo-P3HT) and the functionalized fullerene, [6,6]-phenyl-C_{61}-methyl butyric acid ($PC_{61}BM$). Initially, conformational space was calculated by a systematic grid scan. Then, the lowest energy conformers had their geometry optimized through the Density Functional Theory (DFT) with BLYP functional. Finally, we calculated the interaction potential using the Forcite module available in the Biovia Materials Studio™package (Dassault Systèmes). For this purpose, a code was programmed to rotate both molecules (oligo-P3HT and $PC_{61}BM$) every 5° and 40°, respectively, in the coordinatesx, y, and z. Furthermore, the centroid position of the P3HT oligomer was shifted up to a cutoff distance of 30 Å about the $PC_{61}BM$ for each cartesian axis. For that, oligo-P3HT and $PC_{61}BM$ centroids were initially fixed to equal spatial coordinates. The interaction energy between P3HT:$PC_{61}BM$ complex was calculated for all configurations by applying the Universal force field. Finally, we obtained the configurations with more potent interaction energies, which demonstrate the interaction capacity between of oligo-P3HT and $PC_{61}BM$.

Acknowledgment:

The authors are grateful to the Brazilian research foment agencies FUNCAP, CAPES, CNPq for financial support, and Prof. Valder Freire (UFC) for the support.

References:

[1] LEE, Cheng-Kuang; PAO, Chun-Wei; CHU, Chih-Wei. Energy Environmental Science, v. 4, p. 4124-4132 (2011).

[2] LI, Long-Hua; KONTSEVOI, Oleg Y.; FREEMAN, Arthur J. The Journal of Physical Chemistry C, v. 118, p. 10263-10270 (2014).

[3] WOLF, Caitlyn M. et al. ACS Polymers Au, v. 1, p. 134-152 (2021).

Columnar liquid crystal as aligning host for emissive molecules in OLEDs

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The latest advances with organic semiconductors have provided competitive optoelectronic devices with new functionalities, as observed for OLEDs, OFETs, OPVs and sensors. Liquid-crystalline materials can increase the efficiency of these devices due to their molecular organization that improves charge transport, energy conversion and light outcoupling. In a previous work, we demonstrated that the molecular alignment of a columnar liquid crystal (ColLC) in a diode structure improved the charge carrier mobility in five orders of magnitude and allowed electroluminescence emission [1]. Here, we investigate a triazine-based ColLC (5A) as aligning host for emissive molecules to be applied in the active layer of OLEDs, based on a work where we demonstrated the potential of the photophysical properties of liquid-crystalline triazine-based molecules for alignment-induced enhancement of light outcoupling [2]. As guest emissive molecule we investigated a thermally activated delayed fluorescent compound (CNQxP-TPA), which absorption band overlaps with the emission peak of the triazine-based host in order to optimize energy transfer [3].

Acknowledgements:
INCT/INEO, Capes, CNPq, CAPES-COFECUB (#937/20 and # Ph-C 962/20) and H2020-MSCA-RISE-2017 (OCTA, #778158).

References:
[1] J. Eccher, W. Zajaczkowski, GC Faria, H. Bock, H Von Seggern, W. Pisula, IH Bechtold. ACS Applied Materials Interfaces (Print), 7, 16374-16381 (2015).
[2] André A. Vieira, Giliandro Farias, Wallison C. Costa, Juliana Eccher, Ivan H. Bechtold, Fabien Durola, and Harald Bock. Chem. Eur. J., 27, 9003- 9010 (2021).
[3] Shipan Wang, Yang Miao, Xianju Yan, Kaiqi Ye and Yue Wang. Journal of Materials Chemistry C, 6(25), 6698-6704 (2018).

Combination of titanium dioxide and quinoline derivative thin films as p-n heterojunction inorganic-organic device

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Combinations of inorganic and organic materials have become very attractive for applications in optoelectronics. In this communication, the goal is the assembly of thin film heterojunction of titanium dioxide (n-type semiconductor), with quinoline derivatives, which are organic acceptors (p-type) with blue luminescence [1], with great potential to generate a transparent p-n junction, which can act as gas sensor or photoexcitable optoelectronic device. Tin dioxide was obtained by sol-gel-dip-coating with anatase structure when treated up to 600oC and rutile up to 1000oC. The quinolinic derivative (4-(6-(diethylamino)-4-phenylquinolin-2-yl) benzoic acid was synthed according to the literature [2], in which a reaction occurred between the aniline N,N -Diethyl-p-phenylenediamine and the aldehyde 4-Formylbenzoic acid, forming a Schiff's base, which reacted with phenylacetylene for 4 days under agitation in acid medium, giving rise to quinoline. For the pure form, reactive extraction and recrystallization were performed, followed by solubilization in organic solvents. Homogeneous films have been a challenge due to the tendency to form islands and poor adhesion to the substrate. Drop-casting and electrodeposition processes performed better than dip-coating. Different types of solvents, substrates and annealing are being tested. Confocal microscopy revealed that quinolines solved in acetone deposited via drop-casting have good adhesion and homogeneity, with a thickness of up to 1 micron. UV-Vis transmittance for films of quinoline derivatives 2,4-diphenylquinolin-6-amine and 2-(4-methylthio)phenyl)-4-phenylquinolin-6-amine deposited via drop-casting on FTO substrates reveals absorption bands in the range 360-420nm, in agreement with the literature [1].

Acknowledgments:
FAPESP (Proc. 2018/09235-4, 2022/08483-0 and 2022/12998-5)

References:
[1] G. C. Santos, et al. ; J. Fluor. 27, p. 1709-1720 (2017)
[2] L. M. Martins et al, Orbital: Electron. J. Chem. 14, 1-9 (2021)

Controlling the oxidation state of terbium ions of a borogermanate Tb glass by thermo-electrical imprinting treatment

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Borogermanate glasses containing Tb^{3+}ions are interesting materials due to their luminescent and magnetic properties [1]. The most stable oxidation state of terbium ions is trivalent, but terbium also can present the tetravalent state. Glasses containing these two states can present differences in their structural, luminescent, optical, and magnetic properties, this opens up important opportunities in finding a way to modulate the oxidation state of terbium at the microscale. In this work we used the thermo-electrical poling for the transfer of periodical micro-structured motifs on the surface of a glass with the following composition $96(46GeO_2-29B_2O_3-16Na_2O-5BaO-4Al2O_3)-4Tb_4O_7$. The treatment was performed for 30 min, at 300 °C and a DC bias of 1 kV using an electrode with squared motifs of 40 x 40 μm^2distanced by 10 μm width electrically conductive Pt grid [2]. The presence of molecular oxygen due to the structural rearrangement shows that the poling was effectivein the contact zones. The sample showed a change in color to yellow only in the poled arealocated under the electrode indicating that oxidation of Tb^{3+}ions to Tb^{4+}had occurred. A broad absorption in the visible region between 300 and 650 nm was assigned to the ionsTb^{4+} in the poled layer. The Luminescence measurements showed that a very nice patternwas created in which in the poled zones the luminescence decreases around 10x, while inthe unpoled zones the luminescence was not modified, confirming the control of oxidation of terbium ions at microscale.

Acknowledgements:

The authors would like to thank Brazilian funding agencies FAPESP (2020/15395-4 and 2019/19609-1) for financial support.

References

[1] D. F. Franco, R. G. Fernandes, J. F. Felix, et al, Journal of Materials Research and Technology, 11, 2021, 312.

[2] G. Poirier, L. Karam, V. Rodriguez, et al, Journal of Materials Chemistry C, 10, 28, 2022, 10310.

Degradation study of poly(9,9-dioctylfluorene)-co-trans-stilbene

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Conjugated polymers have been widely studied due to their potential use in the electronics field. One of the key challenges with organic materials is their propensity to undergo photolytic and photochemical reactions upon exposure to light, which can limit their use in certain applications.[1] In many applications, such as in active layers of organic solar cells, photo, and thermal stability are critical factors for material performance. In this context, the goal of this study was to investigate the degradation of the copolymer poly(9,9-dioctylfluorene)-co-trans-stilbene, also known as PFSTB, which exhibits luminescent and semiconducting properties attributed to the conjugated bonds present in its fluorene and stilbene structure. The characterizations involve absorption, emission and structure properties using UV-Vis, fluorescence and infrared spectroscopy, respectively. This material was deposited by blade coater as a thin film, varying thicknesses (one to five layers) on a quartz substrate and subjected to different periods of light exposure (1h to 24 hours). The luminescent properties of the material were measured before and after exposure to light radiation. The UV-Vis technique revealed an increase in the absorption intensity at 380 nm with subsequent layer additions. After exposure to 254 nm light for 24h, the UV-Vis results show no absorption, indicating the degradation of the conjugated polymer. The results obtained contributed to a better understanding of the photodegradation susceptibility of PFSTB.

Acknowledgements:

The authors would like to thank the Federal University of São Paulo (UNIFESP), INEO, CNPq (310647/2020-7), CAPES (Finance Code 001), and FAPESP (2014/50869-6).

References:

[1] WANG, K.; LI, Y.; LI, Y. Challenges to the stability of active layer materials in organic solar cells. Macromolecular Rapid Communications, v. 41, n. 4, p. 1900437, 2020.

Deposition techniques of quinoline-derived organic thin films for coupling with tin dioxide aiming optoelectronic devices applications.

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With the growing interest in organic electronics, quinoline derivatives (QDs) have been investigated for innovation and improvement of optoelectronic devices, being suitable for light-emitting diodes and optical fibers among others [1]. The purpose of this work is to use QDs in the form of thin film, with the premise of their good mechanical stability, resistance to oxidation and p-type electrical transport characteristics [2], and coupling with tin dioxide (SnO2), a naturally n-type semiconductor due to localized donors sites in the matrix. It forms a hybrid organic-inorganic p-n structure, taking advantage mainly of the HOMO-LUMO quinoline electronic transition through its characteristic Donor-π-Acceptor [3] which may be linked to SnO2 characteristics, leading to a luminescent p-n junction. Obtaining a homogeneous film of QDs for application in optoelectronics has proved to be a challenge. Different types of organic solvents have been used to deposit as thin films, by both drop-casting and spin-coating. Best deposition characteristics reveal films ranging from 0.5 μA to 1.0 μA thick and an optical absorption band at approximately 350 nm, which indicates well-deposited film. The influence of different light sources on the electrical response of the QD layer has been investigated, and a He-Cd laser (325nm) irradiated on the device alters its resistivity by up to 3 times. The high transparency in the visible range of the electromagnetic spectrum of tin dioxide as well as of QDs, along with their electrical responses, make the heterojunction promising for the manufacture of organic-inorganic devices.

Acknowledgments:

FAPESP (Proc. 2018/09235-4, 2022/08483-0 and 2022/12998-5)

References:

[1] E. Koścień et al., Opt. Commun. 242, 401-409, (2004).
[2] G. Albrecht et al., Dyes Pigments 158, 334-341, (2018).
[3] G. C. dos Santos et al., J. Mol. Model. 25, 75, (2019).

Designing low-bandgap S,N-Heteroacene oligomers

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Low-bandgap organic molecules are key actors in organic photovoltaic devices. S,N-heteroacenes (SN) are a promising class of such molecules due to their enhanced conjugation compared to S-acenes and acenes [1]. Dicyanovinyl (DCV) is a chemical functional group with high electron affinity that has been shown to decrease the ionization energies and optical bandgap when coupled with S,N-heteropentacenes (SN5' s) and S,N-heterohexacenes (SN6' s) [2]. In this context, we have investigated a series of SN oligomers containing the DCV group in order to understand how one can control optical and transport gap in such systems. These properties were calculated by time-dependent density functional theory (TD-DFT) calculations using the optimally tuned range-separated hybrid functionals (OT-RSH). In the OT-RSH functional the Coulomb operator is split in short and long-range terms, the first one being represented by semi-local approximations and the second one by exact Fock exchange. The range-separating parameter is selected by enforcing that ionization energies equals to frontier orbital eigenvalues (an exact property in the context of Koopman' s theorem). This functional accurately describes charge transfer, making it suitable for studying conjugated systems. It is observed that the DCV-SN oligomers show smaller ionization energies and optical gaps in comparison to their non-functionalized analogous. It depends, however, in the conjugation number of the oligomer. Thus, in our study we show how to design S,N-heteroacenes with the desired optical and transport gap.

References:

[1] C. Huang, X. Liao, K. Gao et al, Chem. Mater. 2018, 30, 15, 5429-5434

[2] C. Wetzel, A. Mishra, E. Mena-Osteritz et al, Org. Lett. 2014, 16, 2, 362-365

Development and fabrication of multilayered core structure silica doped fiber for second harmonic generation

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This work deals with the process of design and fabrication of novel multilayered core structured doped silica fiber through modified chemical vapor deposition (MCVD) for second harmonic generation (SHG). This alternative layered structure has been developed with several fabrication cycles which include a different number of layers (4, 8, and 16) with a dopant such as Germania (GeO_2) into a single mode fiber. These multilayered core structure preforms were characterized in terms of refractive index profiles (RIPs), and weight percent using Wavelength Dispersive Spectroscopy (WDX) and Maker fringe measurements to check their potential for non-linear conversion. Then, drawn into fibers while maintaining their primary geometry. The fibers are characterized in terms of propagation properties and second-order nonlinear (SON) conversion through the coupling of pulsed laser beams into the poled fibers samples. Of these, 16-layered fibers have been demonstrated to produce the most intense SHG resulting in two cycles of fabrication to improve green light emission as detected by a regular camera and a Photomultiplier (1000 mV and 1400 mV) even though it was solely exposed to an external thermal poling.

Development of MEH-PPV Langmuir-Blodgett films for anisotropic devices

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Π-conjugated polymers are widely used as active layers in different optoelectronic devices. However, chain orientation and packing affect charge injection and transport, modifying device performance. The best way to approach this problem is to prepare well-organized films in which the main polymer chains are controlled. In this context, the Langmuir-Blodgett technique becomes an advantageous option, it is possible to obtain an optimization of the organic/inorganic interface, improving charge injection, reducing traps and improving charge transport, generally enhancing devices performance [1,2]. Furthermore, alignment of transition dipole moments will result in polarized absorption and change in emission properties. In this work, we produce Polymeric Light Emitting Diodes (PLED's) using Langmuir-Blodgett films of MEH-PPV to study the correlation between the ordering of the films and device performance. When anisotropic films are used as active layer, we observe that only a fraction of the light (polarized parallel to the polymer transition dipole moment) will be absorbed and the transmitted light portion can be used for other purposes such as absorption by a second active layer. Uv-vis absorption show linear behavior with the number of layers. LB technique introduces partial ordering inducing anisotropic absorption. The dichroic ratio (d) and Huang-Rhys (S) parameter does not show change in solution. In thin films d grows slowly from 1.0 for one layer to 1.3 for 8 layers. On the other hand, S show strong dependence on polarization, indicating that film organization at the interface affects the vibrionic state. Diode devices show polarized light emission.

Acknowledgements:

CNPq, CAPES, FINEP, FAPEPI, UFPI, INEO

References:

[1] OLIVATI, C. A.; et al. Thin Solid Films, v. 520, n. 6, p. 2208-2210, 2012.

[2] VOHRA, Varu ANZAI, Takuya. Journal of Nanomaterials, v. 2017, 2017.

Development of Polyaniline and Lignin PolymericSystems for Application in Supercapacitors

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Developing an efficient energy storage system is crucial for storing energy sources, especially renewable ones that are exponentially increased in the last decade. Supercapacitors have shown significant attraction among the different energy storage systems due to their high-power density, long life cycle, and fast charge and discharge process. Conjugated polymers may be the answer to solve this problem due to their properties, as presented by Polyaniline (PANI). The pseudocapacitive behavior of PANI allows its application in supercapacitor devices with higher power density capacity. In this work, the properties of PANI were combined with Lignin, a biopolymer found in plants and as residue from the pulp and paper industry. Lignin's chemical structure is based on phenylpropane units, which can be oxidized to form interesting products like quinones. These molecules are electrochemically active and can provide two electrons and two H^+ionsper unit, resulting from hydroquinone oxidation into benzoquinone. Herein, PANI-Lignin blends were prepared with variable proportions and tested as electrodes in supercapacitors.The results revealed a capacitance of 518 F g^{-1}at 1 A $g^{-1}$and a retention capacity of 71% after1000 cycles, promising results for developing energy storage devices. The strong interactions between the two materials, provide by hydrogen bonds and π stacking, lead tothe stabilization of the structure of PANI. These bonds act as a bridge to control the volumechange during the charge/discharge process that naturally happens and causes PANI degradation, promoting improved mechanical and electrochemical properties. Therefore, this study shows a low-cost and eco-friendly solution combining industrial recycling wasteand further application in energy storage, creating a potential circular economy through green chemistry.

Acknowledgements:
INEO, CNPq, Fundação Araucária.

Diketopyrrolopyrrole derivatives: influence of peripheral groups in the excited states absorption process

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Organic π-conjugated molecules have emerged as promising candidates for applications in nonlinear optics, such as optical limiting, optical switching, fluorescent probes, among others, due to the ease of structural modification, which allows for exploring more efficient molecular designs at a relatively low production cost [1]. Among the various organic materials studied, diketopyrrolopyrrole (DPP) derivatives stand out for their high structural planarity and good emissive properties, making them excellent candidates for nonlinear optics research [2]. Here is reported the study of excited state absorption (ESA) in two diketopyrrolopyrrole derivatives containing thienyl moiety and different peripheral groups (hydrogen and bromine) employing the Z-scan and pump-and-probe techniques. It is expected that the structure containing the bromine atom will exhibit an increase in the nonlinear response owing to the higher induced polarizability by the bromine. The results obtained by the Z-scan technique revealed an ESA band around 415 nm, with an excited state cross-section (σ_1n) about 15 times larger than the ground state. In addition, the sample containing the bromine atom showed an approximately 2-fold increase in σ_1n. On the other hand, by the pump-and-probe technique, another ESA band at around 750 nm was observed. These results provide a conceptual proof that diketopyrrolopyrrole derivatives are promising materials for applications in photonic devices.

[1] Cocca et al., J. Photochem. Photobiol. B Biol., v. 175, p. 1-8, 2017;
[2] Scalon et al., ACS Appl. Polym. Mater., v. 3, p. 4223-4233, 2021.

Dynamics of the Generation and Propagation of Independent OAM Modes in a Photonic Chip

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The remarkable features of the intensity distribution and phase of the orbital angular momentum (OAM) beams have been used for modulating classic and quantum information. Although there is a broad and rich ground of works investigating the free propagation of the structured beams it is still a challenge to incorporate their many degrees of freedom within compact and integrated photonic devices. Recently, Y. Chen et al. [1,2] took an important step forward in this direction, but limited to the OAM modes that can be guided in each structure. Starting at this point, we demonstrated that it would be possible to guide different OAM modes in the same photonic structure. By means of a simple coupling system we could tune the output OAM mode by properly tilting the input beam [3]. The proposal presented here consists of the microfabrication of annular waveguides inside a glassy blade via direct laser writing technique. We understood that the adequate phase matching phase, between the input beam and the Δ parameter, selects the OAM beam modes that will be found in the output waveguide. The results confirmed that a tilted input coupling can selecte propagate different OAM modes and its superpositions inside the photonic chip with high purity. Owing to the degree of freedom of OAM light, OAM circuit photonics can be explored to enhance high- capacity data processing for quantum or classical networking.

Acknowledgements:

The present research was supported by Fundação de Amparo à Pesquisa do estado de Alagoas (FAPEAL), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), and Coordenação de Aperfeiçoamento de pessoal de Nível Superior, Finance Code No. 001(CAPES).

References:

[1] Y. Chen, et al. Physical Review Letters 121, 233602 (2018).
[2] Y. Chen, et al. Physical Review Letters 124, 153601 (2020).
[3] Jonathas M. Oliveira, et al. Physical Review A 104, 6 (2021).

Effect of aging of latent fingerprint residues developedby PEDOT on metallic surfaces

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The aging of fingerprints is a major challenge for forensic science because of the progressive degradation of organic compounds under the influence of several factors: type of suspect latent print residue, texture and condition of surfaces (clean, dirty, sticky, greasy etc.), environmental conditions during and after latent print deposition, and time elapsed since the evidence was touched [1,2]. This work evaluates the effect of the aging of the residues present in the fingerprint on the quality of the images revealed through the electrochemical deposition of poly(3,4-ethylenedioxythiophene) (PEDOT) on stainless steel and cartridge cases. PEDOT was deposited electrochemically by chronoamperometry applying an electrical potential of 0.9 V *vs.*Ag/AgCl for 180 s on stainless steel and cartridge case surfaces. The images of the fingerprints revealed from the electrodeposition of the PEDOT were registered after 1, 7 and 30 days resulting from the application of the fingerprint on the metallic surface. Fingerprint developments show the aging effect of latent fingerprints developed through PEDOT electrodeposition on stainless steel plates and cartridge cases. The contrast between the fingerprint and the metallic surface is noticeable in fingerprints aged for up to 30 days. Papilloscopic details such as line ends, bifurcations and pores were found. This result shows that the amount of sebaceous material present in the fingerprint directly influences the deposition of the PEDOT, since the fat is acting as an inhibiting barrier and indicate that processes involving the electrodeposition of PEDOT on metallic surfaces consist of an efficient methodology to reveal latent fingerprints, even after 30 days in which the sebaceous/eccrine residues were left on the surface.

Acknowledgements:
CNPq, CAPES, FAPEAL, INC Federal Police.

References:
[1] Costa CV et al. Synthetic Metals v.262 p. 116347 (2020).
[2] Costa CV et al. Nano Select v. 1, p. 1-8 (2020).

Effect of TeO2 on optical and 1.06 µm emission in Nd3+-doped phosphate glass for high-power lasers

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In this work, we deal with glass composition, $29P_2O_5$+(60−□)ZnO+$10Na_2O$+xTeO_2(□=5−40mol.%) doped with 1 mol.% of Nd_2O_3. The samples are characterized by x ray diffraction, micro-Raman, optical absorption, and photoluminescence techniques. Refractive index (□) linearly increased with TeO_2content, as well as the optical basicity (Λ) due to the strengthening of the covalent bond between cation and oxide ions. Such information was corroborated by Raman spectra via an increase of non-bridging oxygens. The maximum phonon energy is approximately 1010cm^{-1}, which is lower than reported for P_2O_5−ZnO−Na_2O phosphate glass (1164cm^{-1}). Detailed spectroscopic and luminescent properties based on TeO_2content are discussed. From luminescence spectra, laser parameters were estimated, viz., radiative transition probabilities, radiative lifetimes, branching ratios, and emission cross sections. The results show that the optimal laser emission parameters are obtained for TeO_2content equal to 20 mol %, which evinces this glass composition as a promising host material for 1.06 µm high-power laser applications.

Effect of the side chain in diketopyrrolopyrrole copolymers: a comparative study between hydrophobic alkyl and hydrophilic triethylene glycol chains

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Organic materials, and particularly conjugated polymers, have played an essential role in optoelectronics. In recent years, considerable attention has been devoted to organic dyes, such as diketopyrrolopyrrole (DPP). In this molecule, changes in the chemical structure can easily modulate their electronic and optical properties. In contrast to the great efforts to develop new conjugated polymer backbones, there are few studies involving the structure-properties correlation of conjugated polymers containing polar side chains, mainly involving electrochemical techniques. In this work, the effect of the DPP side chain (triethylene glycol and hexyl) on the electrochemical properties of a donor-acceptor (D-A) copolymer containing fluorene and DPP units, connected through thiophene rings, has being studied. The cyclic voltammetry technique was further employed to investigate the electrochemical behavior and the stability of the copolymers. For the copolymer with alkyl side chains, four quasi-reversible redox peaks were observed at 1.37 V/1.06 V, 1.56 V/1.34 V, 0.90 V/-0.67 V, and - 1.29 V/-1.16 V (vs NHE), which are associated with the p and n doping. The p-doping/dedoping processes did not show a significant drop in the current peak response of the copolymer even after 410 cycles. By contrast, the reduction process was seen to be quite unstable, and the loss of electrochemical activity occurs already in the 10th cycle of n-doping. Concerning the copolymer with glycol side chain, the preliminary results pointed out a better stability for the p and n doping, probably due to its ability to effectively take up ions from the supporting electrolyte at a relatively fast rate. This electrochemical stability is quite interesting for the application of this polymer as p or n-type material in supercapacitors, smart windows, electrochemical transistors, or even in energy storage devices such as Li-ion batteries.

Acknowlegments:
INEO, CNPq, Fundação Araucária.

Effect of WO_3 in the nonlinearities properties oftungsten lead pyrophosphate glasses

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Properties such as high chemical stability, photochromism and efficient up conversion [1,2,3], made glasses with phosphate and tungsten matrix potential candidates for optical applications. In this direction, studies with the $NaPO_3$-WO_3 binary vitreous system made it possible to correlate, among other aspects, the increment of tungsten trioxide with the formation of clouds with WO_6 units - highly polarizable, promping the search for compounds with similar structural properties and optimized nonlinear optical responses. In this work, the linear and nonlinear optical responses of the $Pb_2P_2O_7$−WO_3 glass matrix will be characterized due to its structural similarities with the $NaPO_3$-WO_3 system. Yet, the replacement of sodium by a heavier atom results in extended transparency [4] and possibly in higher nonlinearities. For the sample characterization we applied UV-Vis absorption, photoelectron spectroscopy (XPS), and the refractive Z-Scan technique. These techniques make it possible to obtain linear parameters (absorbance, linear refractive index, and chemical conformations) and the dispersion of the nonlinear refractive index, in addition to providing conclusions about the origin of the nonlinear response of the matrix. Finally, high nonlinear refractive responses were observed, paving the way for the use of these compounds in telecommunications and as possible photonic devices.

References:

[1] POIRIER, G. et al. New tungstate fluorophosphate glasses. J. Non-Cryst.Sol. p. 293-298, 2005.
[2] POIRIER, G. et al. Photochromic properties of tungtate-basedglasses. Solid State Ionics, v. 178, p. 871-875, 2007.
[3] POIRIER, G. ; JEREZ, V. A. ; ARAÚJO, C. B. de. Optical spectroscopy and frequency up-conversion properties of Tm 3+doped tungstate fluorophosphate glasses. J. Appl. Phys. v. 93, n. 3, p. 1493-1498, 2003.
[4] MANZANI, D. et al. Thermal, structural and optical properties of new tungsten lead-pyrophosphate glasses - Opt. Mater. v. 33, n. 12, p. 1862-1866, 2011.

Electrochemically doped melanin films with improved conductivity for bioelectronics application

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Bioelectronics combines electronic and biological components in order to develop miniaturized implantable devices capable of altering and controlling electrical signals in the human body. Eumelanin is a natural pigment, biocompatible composed of different structural subunit which can be particularly valuable in the field of bioelectronics because it is a hybrid-type conductor [1]. However, the low conductivity of melanin limits the devices development [2]. In this work, electrochemical doping was proposed to increase the electronic and ionic conductivity of different synthetic melanin derivatives. Melanin films were prepared and electrochemically treated using a three-electrode cell. Different electrochemical parameters were evaluated (scan rate, potential windows and electrolyte concentration). Current-voltage and electrochemical impedance spectroscopy measurements showed that the electronic conductivity of the doped melanin increased when compared to melanins without electrochemical treatment. The experiments were complemented by others chemical characterization techniques, indicating the transfer of species from the electrolyte solution to the melanin film. Our results suggest that electrochemical doping could be an alternative to increase the conductivity of melanin films, allowing its use in the manufacture of organic electrochemical transistors (OECTs).

Acknowledgements:
FAPESP (2021/09207-3) and LNNano-CNPEM

Reference:

[1] Mostert A. B., et al. Sci. Adv. 4, 1293 (2018).
[2] Eon T, et al. Part. Part. Syst. Charact. 36, 1900166 (2019).

Electronic effects of N- and S- functionalization on Hydrogenated Diamond Surfaces

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Diamond surface functionalization has received significant research interest recently due to the impact on the surface electronic structure, which is of interest in engineering diamond as a semiconductor. To understand the modifications on diamond surface electronic structure caused from sulfur and nitrogen groups, we performed Density Functional Theory (DFT) simulations using a PAW/GGA approach [1], as implemented in the computational code Quantum Espresso (QE) [2]. We explored 6 different surface coverage: 100%H, 91%H/9%S, 92%H/8%NH_2, 83%H/9%S/8%NH_2, 74%H/18%S/8%NH_2, and 75%H/9%S/16%NH_2. Our results indicate that the diamond surface functionalized with S or/and NH_2presents a lower the bandgap in comparison to the 100%H, which is caused by the electronic levels that came from S and N atoms. The S atoms produces a new conducting band minimum lower than that observed for the 100%H, and the N atom could increase slightly the valence band maximum. The best results for the bandgap energy are seen for the 75%H/9%S/16%NH_2surface coverage, once the S atom keep the conducting band minimum lower than that for 100%H and the percentage of NH_2higher than S can affect the valence band maximum increasing it in energy, resulting in a decreased bandgap. The results show that the S determine the conducting band minimum, but It is important to keep the percentage of NH_2higher than S on the surface to guarantee the increasement of valence band maximum, since the S could keep it lower.

References:

[1] Perdew, J. P.; Burke, K.; Ernzerhof, M. Generalized Gradient Approximation Made Simple. Phys. Rev. Lett. 1996, 77 (18), 3865-3868.
[2] Giannozzi, P. et al. QUANTUM ESPRESSO: A Modular and Open-Source Software Project for Quantum Simulations of Materials. J. Phys. Condens. Matter 2009, 21 (39), 395502.

Energy transfer processes in BBPT glasses doped with Nd^{3+}

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Solid-state lasers operating in the visible and near-infrared (NIR) regions are currently in focus due to their potential applications in technology, telecommunications, sensors, defense and diagnostics in medicine. In the present work, we analyzed boroteluride glasses (B_2O_3+Bi_2O_3+PbO+TeO_2) doped with Nd^{3+}ions, prepared by cooling melting and characterized by optical absorption techniques (UV-Vis-NIR-MIR), near infrared emission. The decay of Nd^{3+}: $^4F_{3/2}$were characterized by Judd-Ofelt parameters (Ω_λ, $\lambda = 2$, 4 and 6), radiative transition probabilities (A_{rad}), radiative lifetimes (τ_{rad}) and branching ratios (β). The Judd-Ofelt parameters were obtained from the emission and absorption spectra. With these parameters, it was observed that the matrix did not undergo a significant change as a function of the concentration of 0.5Nd^{3+}ions.

Acknowledgements:

The authors acknowledge the financial support of Brazilian funding agencies CNPq, UFJF, CAPES and FAPEMIG.

Reference:

[1] M. Seshadri, V. Anjos, M.J.V. Bell, Energy transfer process and radiative properties of 1.06 µm emission in Nd^{3+}doped TeO_2-ZnO-Na_2O glasses Journal of Luminescence, 196 399-405 (2018).

Engineering nano and natural-form of conjugated copolymer thin films with silver nanoparticles: structural and morphological studies

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The electrical, thermal, and optical properties of conjugated polymers (CPs) and metallic nanoparticles make them interesting components in the development of devices, because of the formation of nanocomposites with improved properties [1]. However, the hydrophobicity property of CPs can hinder their application in aqueous media. Recent studies demonstrated that this can be overcome by the formation of CPs nanoparticles in aqueous media [2]. Thus, this work characterizes the morphological and structural properties by AFM, XPS and Raman spectroscopy of thin films composed of the copolymer poly(9,9-dioctylfluorene-co-3,4-ethylene dioxythiophene) (PDOF-co-PEDOT) in its nanoparticle (NCP) and natural form, combined with silver nanoparticles (AgNP) of different morphologies (spherical and triangular), aiming further application of these nanocomposite films as SERS-active layers for pesticide sensors. AFM images of NCP/AgNP films showed specific structures that were transferred to the solid substrate. The films' morphology could also be influenced by the geometry of AgNP. XPS data demonstrated that the insertion of the AgNP can result in the oxidation of the polymeric chains, but the discovery that NCP films are more resistant to this type of degradation makes this material more interesting for future applications. Raman spectra showed the enhancement effect of polymer Raman bands, indicating the SERS effect induced by the AgNP. Due to these interesting properties, these specific films can be promising in future applications as SERS sensors, as well as in applications where degradation processes are important to be considered.

Acknowledgments:

We gratefully acknowledge CAPES nº 88881.689895/2022-01, FAPESP (2014/50869-6) and CNPq (310647/2020-7).

References:

[1] M.M.Ayad, N.Prastomo, A.Matsuda, J.Stejskal, Synthetic Metals, 160, 42 (2010)
[2] Y.Braeken, S.Cheruku, A.Ethirajan, W.Maes, Materials (Basel), 10, 1420 (2017)

Evaluating the impact of solvent vapor annealing in organic thin films through transfer matrix method

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In this work, a solvent vapor annealing procedure (SVA) was used to improve the optical and adhesion features of a conjugated copolymer:molecule acceptor film. The power dissipation was simulated along the structure of organic photovoltaic device devices considering the interference occurring in stacked thin films under normal incidence by using the transfer matrix method (TMM). Therefore, the monitoring through ellipsometry and TMM modelling contribute to understand the behavior of the electromagnetic wave inside the device, providing insights about proper optimizations that can be performed to increase the G(x) rate and the Jsc parameter. This model takes into count the experimental values of refractive index n and extinction coefficient k acquired from the D:A film to simulate the spatial distribution of the electric field and provide valuable information about photovoltaic parameters. The results pointed out that the SVA-treated films presented distinct refraction index curve, as well as improved electric field distribution, when compared with cast films.

Evaluation of stability and bioactivity of a nanocomposite formed by conjugated polymer andsilver nanoparticles

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One of the great advantages of mixing components in a single system, in addition to using smaller amounts of samples, is that the properties arising from the isolated materials are gathered in a single final product [1]. In this scenario, we propose the combination of conjugated copolymer nanoparticles (CPN) composed of poly(fluorene/thiophene) with spherical silver nanoparticles (AgNP) in different proportions (v/v) (CPN:AgNP_1:5, 1:3, 1:1 and 3:1) aiming to obtain a stable nanocomposite with bioactivity against microorganisms such as bacteria. Therefore, aqueous dispersions of both materials were prepared and analyzed by UV-Vis absorption spectroscopy, fluorescence, dynamic light scattering (DLS), and zeta potential, in order to evaluate the samples stability, during three weeks after preparation. All samples demonstrated stability for at least two weeks. Two samples in different ratio (CPN:AgNP_1:3 and 3:1), as well as the isolated components, were selected for minimum inhibitory concentration (MIC) analysis, against the bacterial Escherichia coli and Staphylococcus aureus. The bioassay results showed enhanced bioactivity in the composites compared to those of prevenient from metallic silver nanoparticles or CPN alone. Prominent results were obtained using the nanocomposite CPN:AgNP_1:3. In this sense, it was possible to conclude that nanocomposite preparation could efficiently improve nanocomposite stability and microbicide properties, which augurs well for further developments of CPN/AgNP microbicide materials.

Acknowledgment:

We are thankful to INEO, Capes (Finance Code 001), and FAPESP (2014/50869-6).

Reference:

[1] M. Z. Strzalka, A. D. Marczewska, Int J Mol Sci, 21 (24), 9388, 1-28 (2020).

Evaluation of the Structure-Property of Thin Films of Polyaniline-based Nanocomposites with Metal Oxides Deposited via Spin Coating

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Polyaniline (PANI) has a special position in the field of conducting polymers due to its simple and low cost synthesis, high conductivity and stability, deposition as thin film and special proton doping mechanism. Thereby, this polymer is a candidate for many applications, such as capacitors, chemical, gas and biological sensors [1]. PANI properties can be shaped upon exposure to acids and bases, and when incorporated with different materials like metal oxides. Its combination with metal oxides presented promising results for electrochemical sensor applications. As shown in our previous work [2], combining PANI with metal oxides improved the sensor''s performance in linearity and sensitivity. The present work pursues improving the previous approach and seeks to produce low-cost electrochemical pH sensors using thin nanocomposites films of PANI and metal oxides. We use PANI/CeO2 and PANI/WO3 nanocomposites, deposited via spin coating. The films were produced using a PANI dispersion and a metal oxide (CeO_2or WO_3) solution, both in N-methyl-2-pyrrolidone (NMP, 99%) mixed in five different proportions (90/10, 70/30, 50/50, 30/70, 10/90, V/V) [3]. The films of PANI/$CeO_2$and PANI/WO_3were applied in the electrochemical impedance and capacitance spectroscopy (EIS and ECS) transduction stages as pH sensors, and characterized by their thickness and roughness, UV-VIS absorbance spectra. The structure-property relationship of the nanocomposite thin films is dependent on the proportion of its constituents and directly influences the sensor's response.

Acknowledgements:

Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) and Fundação deApoio à Pesquisa.

References:

[1] Babel, V., Hiran, B. L. Polymer Composites, 42(7), 3142-3157 (2021)

[2] Rossignatti, B. C., Vieira, A. P., Barbosa, M. S., Abegão, L. M. G., Mello, H. J. N. P. D. Polymers, 15(3), 578 (2023)

[3] Mello, H. J. N. P. D., Mulato, M. Journal of Applied Polymer Science, 135(33), 46625 (2018).

Experimental and theoretical characterization of adenosine hydrochloride crystals

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Crystallography is a broad area of study that incorporates studies of organic and inorganic materials of biological interest. This work studied the crystal of adenosine hydrochloride (AC). AC crystals were grown, characterized by X-ray diffraction (XRD), differential scanning calorimetry (DSC) and Raman spectroscopy, Hirshfeld surface and their vibrational Raman spectra were calculated and compared to the experimental, in addition to the frontier molecular orbitals. XRD data refined by the Rietveld method show that the crystal belongs to the monoclinic system, P21-space group, and lattice parameters with values similar to the literature [1]. In the DSC analysis, it was identified that no thermal event occurs before the melting of the material, which occurs around 145 ºC. Hirshfeld surface analysis shows the crystal packing interactions and their importance, as well as the relative amounts of each interaction in the 2D fingerprint. The calculated geometric data showed a lot of similarity with the experimental data, as well as the Raman spectra showed good agreement between theoretical and experimental results. The gap energy (Egap) was 5.47 eV, with the border orbitals present in greater density in the adenine portion of the AC molecule, with an evident difference in the chlorine atom, fully occupied by electronic density in HOMO. Egap value of the AC molecule was higher when compared to 5mU, showing that AC is probably less reactive than 5mU [2], as shown by chemical hardness.

Acknowledgment

We thankful the CNPq, CAPES, FAPEMA, PPGCM-UFMA, LER and computational support of CENAPAD - SP (proj780).

References

[1] K. Shikata, T. Ueki, T. Mitsui. Acta Crystallogr B. 29 (1973) 31-38.

[2] J.O. Carvalho, J.G. Oliveira Neto, J.G. Silva Filho, F.F. de Sousa, P.T.C. Freire, A.O. Santos, P.F. Façanha Filho. Spectrochim Acta A Mol Biomol Spectrosc. 281 (2022) 121594.

Field-effect sensor based on $CoFe_2O_4$ nanocrystals and graphene oxide as a sensitive layer for environmental applications

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Sensor devices have shown significant potential in portable microelectronic systems for biomedical and environmental applications. To obtain sensing units with superior properties and performance, it is crucial to study (nano)materials and their optimal integration in the device as a receptor layer for the target analyte [1]. In this context, cobalt ferrite ($CoFe_2O_4$) has been reported as an attractive material for H_2O_2 catalysis, as well as graphene oxide (GO), which has unique properties for H_2O_2 detection and heavy metal adsorption [2]. This study was aimed at investigating the effect of layer-by-layer (LbL) films containing $CoFe_2O_4$ nanocrystals embedded in PAH and GO as a receptor layer (sensing unit) on an electrolyte-insulator-semiconductor (EIS) field-effect sensor platform. LbL films with PAH-$CoFe_2O_4$/GO structure were applied to detect H_2O_2, Cd^{2+}, and Cu^{2+} ions, envisaging a proof-of-concept field-effect sensor for environmental applications. Scanning electron microscopy (SEM) revealed a LbL film morphology with high surface area featuring heterogeneous clusters of nanocrystals covered by a homogeneous GO coating. Electrochemical characterizations were carried out by capacitance-voltage (C/V) and constant-capacitance (ConCap) measurements demonstrating catalytic characteristics in detection experiments of an optimized EIS-LbL sensor containing 6 bilayers. This sensor system was sensitive and exhibited a low limit of detection of ca. 314 µM for H_2O_2 and 0.54 µM and 0.47 µM for Cd^{2+} and Cu^{2+} ions, respectively. These results prove the relevance of the PAH-$CoFe_2O_4$/GO structure as a receptor layer in EIS devices for environmental applications.

Acknowledgements:

CNPq (Grants 152155/2022-8 and 437785/2018-1) and FAPEMIG (Grant APQ-01464-18)

References:

[1] R. Ding, Y. H. Cheong, A. Ahamed, G. Lisak, Anal. Chem., 93, 1880-1888 (2021)

[2] P. V. Morais, A. C. A. Silva, N. O. Dantas, M. J. Schoning, J. R. Siqueira Jr., Phys. Status Solidi A 216, No. 1900044 (2019)

Fluorescent polymer films based on thiophene and fluorene with electrochromic properties

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Conjugated polymers have attracted broad academic and industrial interest due to their applications in polymer light-emitting diodes, polymer solar cells, electrochromic devices and fluorescent sensors. Recently, multifunctional stimuli-responsive optical materials showing electrochemical switching of both coloration and emission based on conjugated polymers have been synthed through incorporation of electroactive fluorophores into the polymer main chain and their properties have been investigated aiming at application in electro-optical devices [1]. Fluorene and polyfluorene derivatives have a rigid planar structure, excellent hole-transporting properties, chemical stability and photoluminescence efficiencies. Thus, the preparation of extended π-conjugated systems based on the introduction of a fluorene bridge into the bisthiophene main chain can render multifunctional materials that exhibit both electrochromic and fluorescent properties. Herein a p-extended system based on thiophene and fluorene, named 2,2’-(9,9-dioctyl-9H-fluorene-2,7-diyl)bisthiophene (FBT), was electrodeposited on Indium Tin Oxide (ITO) electrodes at relatively low potential and the electrochromic properties of its polymer films (PFBT) were investigated. The changes in the spectral absorption bands according to the potential applied to the system were interpreted by using the CIE 1931 xy chromaticity coordinates. The colors of the polymer film change from yellow in the neutral state (E = 0.00 V) to green in the oxidized state (E = 0.85 V). Simultaneously, the PFBT films are fluorescent in the neutral state and the emission is quenched at anodic potentials. Thus, the high electrochromic and fluorescence contrast of the PFBT films according to their oxidation states makes them suitable for application in multifunctional optical devices, such as electrofluorescent devices.

Acknowledgements:
CNPq, CAPES, FAPEAl, IFAL

References:

[1]NOGUEIRA, FAR et al. J. Braz. Chem. Soc, v. 30, p. 2702-2711(2019)

Functionalized hybrid materials for photonics and biological systems

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Functionalized hybrid materials have unique physicochemical properties and currently are employed in several technological fields, since optics and photonics, until biomedical applications. For instance, a hybrid matrix functionalized with optically active compounds allows potential materials for RGB-devices fabrication or random laser systems, due to their convenient optical and morphological characteristics. Additionally, such matrices allow the fabrication of specific photonic devices with nonlinear responses, such as optical limiters. Regarding biomedical applications, hybrid materials can be sculpted using high intensity lasers and miniaturized devices can be produced for desired systems. In this context, the present work shows the synthesis of hybrid matrices functionalized with different optically active compounds, such as organic dyes, lanthanide complexes, semiconductor nanoparticles or crystalline forms of Carbon, in order to produce samples for photonics experiments. The mixture of a silane compound and acrylate monomers composes the host matrix. Using these functionalized matrices, we produced thin films and bulks, obtaining structures like *xerogel* or *aerogel*. Such samples presented structures with thermal stability and high mechanical resistance, among other interesting features for prospect applications. We characterized the prepared samples using several methodologies, such as optical spectroscopy, photoluminescence and electron microscopy. Finally, we also fabricate miniaturized devices using direct laser writing (DLW) technique, which employ femtosecond lasers, with high intensity pulses. The fabricated devices have micrometer resolution and are potential structures for photonics and biological applications.

Acknowledgments:

The authors acknowledge the financial support from PROPe UNESP (Pró-Reitoria de Pesquisa da UNESP), CNPq, CAPES and FAPESP.

Immobilization of graphene oxide and laccase on lipid Langmuir-Blodgett films: possible application for biosensors for phenolic compounds

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Inserting graphene oxide in Langmuir-Blodgett (LB) films of enzymes confined in lipids can improve their catalytic performance, which is interesting for bioelectronics since robust biosensors can be assembled. In this work, we incorporated the enzyme laccase and graphene oxide from the aqueous subphase in 1,2-dimyristoyl-sn-glycero-3-phosphatic acid (DMPA) Langmuir monolayers and transferred them to solid supports as LB films to essay the catalytic activity towards the oxidation of a variety of phenolic substrates, performing the electronic transfer. Surface pressure-area isotherms, polarization-modulated infrared reflection-absorption spectroscopy, and Brewster angle microscopy characterized the floating monolayers. The adsorption of the enzyme at the air-water interface condensed the lipid monolayer, altering the film compressibility at high surface pressures. The monolayers were transferred to solid supports as LB films and characterized with fluorescence spectroscopy and quartz crystal microbalance. Viability as sensors was tested with UV-vis spectroscopy. We believe that the enzyme accommodated in the DMPA LB films preserves the catalytic activity, and graphene oxide helped conserve the catalytic properties for more extended periods of storage. The method presented in this work allows for controlled catalytic activity and can help explain why specific film architectures exhibit improved performance.

Acknowledgments:
FAPESP and CNPQ

Impact of Microwave-Assisted Synthesis on the Triboluminescence of Europium Complexes with β-Diketonate Ligands

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Triboluminescent materials, such as crystals of europium complexes, convert mechanical energy into visible light, making them attractive for potential applications in the development of real-time structural damage sensors [1]. Preparation methods have been shown to have a significant impact on the triboluminescence properties. For example, when [Eu(DBM)$_3$(TPPO)$_2$] was prepared by a conventional heating synthetic methodology, as it has already been reported in the literature, apparently that led to no triboluminescence [2]. In this work, we demonstrate that the use of microwave-assisted syntheses can lead to triboluminescent crystals of europium complexes with β-diketonate ligands. The microwave reactor heating is uniform and impacts the crystallization of these compounds. Accordingly, we prepared the ternary europium (III) complexes [Eu(β)$_3$(TPPO)$_2$] and [Eu(Cl)(β)$_2$(Phen)$_2$] with the ionic β-diketonate ligands (β = DBM, BTFA, TTA, TFA, HFAC) and the non-ionicligands TPPO (triphenylphosphine oxide) and Phen (1,10-phenanthroline). The optimizedsynthetic conditions were 100 W, 75 °C during 10 minutes for each reaction step. Theproducts were characterized by FTIR and NMR (^{1}H and ^{19}F) spectroscopies. As a result, all these ternary complexes were found to exhibit luminescence upon mechanical stress (rubbing), confirmed by acquiring their triboluminescence spectra, which showed transitions that are typical of the europium ion. Based on our findings, we conclude thatutilization of the microwave-assisted method for the preparation of triboluminescent materials has significant potential for further development of more responsive smart materials and structural damage sensors.

Acknowledgements:
This work was supported by CNPq, PRONEX/FACEPE and CAPES.

References:

[1] J. G. Bünzli and K. Wong, Journal of Rare Earths 36, 1-41 (2018).
[2] N. B. D. Lima, A. I. S. Silva, S. M. C. Golçalves and A. M. Simas, Journal of Luminescence 170, 505-512 (2016).

Investigating optical non-linearities and triplet quantum yield in porphyrins-chalcone structures

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Porphyrin's molecular structure confers the ability to efficiently generate singlet oxygen, which is the primary cytotoxic agent responsible for the elimination of cancer cells, and this efficiency is related to the formation of molecules at the triplet long lifetime excited state. Organic molecules exhibiting nonlinear optical responses have key advantages because of their potentially increasing depth of light penetration in tissue. In this work, to complement the triplet study, we report on the study of two-photon absorption (TPA) spectra of a series of tetraphenyl porphyrin with chalcone derivatives groups in its neutral and cationic forms. It was expected that the electronic nature of porphyrins leads to a significant change in two-photon absorption cross-sections due to different electron-accepting groups and symmetry changes, influencing the TPA magnitude with distinct chalcone derivatives, which is extremely important in applications such as fluorescent bioprobes. In this way, the two-photon absorption cross section peaks exhibited values in the range of 15 GM in porphyrin'sQ bands, which is a terapeutic window, interesting for biological applications and not so much found in literature.

As it is known, so far, in the literature a few works report measurements on TPA cross-sections in porphyrins molecules, which is known to be difficult because of the easy aggregation of those at low molar concentrations. Thus, to circumvent this problem, we have set up a technique based on monitoring the fluorescence induced by a two-photon absorption that can be used for solutions at very low concentrations, avoiding aggregation.

Investigation of nanostructured films containing PAMAM, reduced graphene oxide and tungsten oxide for application in supercapacitors

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Supercapacitors are energy storage devices that have attracted attention of researchers due to their unique characteristics, including as high power density, fast charge and discharge rates and long lifetime [1,2]. Different combinations of materials and different ways of arranging them have been the focus of research in recent decades. In this study, we investigated the combination of poly(amidoamine) (PAMAM), reduced graphene oxide (rGO) and tungsten oxide (WO3) as nanostructured films assembled on conductive substrates of indium tin oxide (ITO) as a supercapacitor electrode for energy storage application. Using the layer-by-layer technique, multilayer films with high capacitance values were found for films containing 20 bilayers. Cyclic voltammetry revealed an areal capacitance of ca. 6.5 mF/cm² at a scan rate of 1 mV/s. The galvanostatic charge-discharge characterization pointed to a discharge time of ca. 300 s at a current of 3 µA, which permited reaching aspecific capacitance value of 75 F/g, with an energy density of 5 Wh/kg and a power densityof 60.6 W/kg. The latter values compared with blank control electrodes without WO3 exhibited capacitance and energy and power densities 2.5 times lower, demonstrating thatthe presence of WO3 was essential to achieve a supercapacitor with improved performance. These findings prove that nanostructured films based on rGO-WO3 can be employed andexpanded for supercapacitors applications.

Acknowledgements

The authors are grateful to the Brazilian Foundations FAPEMIG (Grants APQ-01464-18 and BPD-00880-22) and CNPq (Grants 437785/2018-1 and and 150431/2023-6).

References

[1] XIAO, H. et al. Journal of Alloys and Compounds, vol. 738, p. 25-31 (2018)

[2] JI, J. et al. ACS Nano, vol. 7, p. 6237-6243 (2013)

Investigation of naphthalenediimide with phenothiazineas photoinduced charge-separating junction

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Photoinduced charge separating molecular systems are often key players in clean energy generation, such as organic photovoltaic devices.[1] We investigated π-junctions of N,N'-bis(3-triethoxysilylpropyl)-1,4,5,8-naphthalenediimide (NDI-silane) with 3,7-di-t-butylphenothiazine (TBP) as donor (D) - acceptor (A) system for photoinduced charge separation. From Time-Dependent Density Functional Theory (TD-DFT) calculations with optimized hybrid functionalization (OT-RSH), we observed in the NDI-TBP dimer and the NDI-TBP multimolecular configuration an alignment of the HOMO and LUMO eigenvalues of the molecules, in a "cascade fashion". As a consequence, the NDI TBP-dimer and a multimolecular configuration show excited states with a strong charge transfer character, around 1,4 eV (O.S ≈ 0.015) and 1,0 eV (O.S ≈ 0.013), respectively. In the experimental study, we first derived a methodology to obtain pure and stable crystals of NDI-silane from crystallization in petroleum ether and ethyl ether with a net yield of 10%,[2] confirmed by liquid state ^{1}H, ^{13}C and ^{29}Si nuclear magnetic resonance spectroscopy. This precursor was used to prepare NDI-silane and a 1:1 v/v NDI-TBP thin films by spin coating. They were characterized by optical and atomic microscopy, profilometry, absorption spectroscopy, and photoconductivity. The films were obtained with high homogeneity and an optimal thickness of 300-500 nm, demonstrating significant absorbance in the UV and blue spectral region, and a measurable photo-conductivity effect. In conclusion, it was noted that the NDI-TBP heterojunction demonstrates great potential to be applied as photoinduced charge-separating systems.

Acknowledgments
We acknowledge FAPESP and the PRH-ANP UFABC program for financial support.

References

[1] H. Spanggaard e FC Krebs, Sol. Energ. Esteira. Sol. C. 83, 125 (2004).
[2] B. Castanheira, et al., Langmuir 34, 8195 (2018), pMID: 29909638, DOI: https://doi.org/10.1021/acs.langmuir.8b00220

Layer-by-Layer (LbL) films of DODAB/NiTsPc applied to detect carbendazim (MBC) pesticide by impedance spectroscopy

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Usually, electronic tongues (e-tongue) do not require specific interactions to discriminate aqueous solutions. Among the several factors which determine the electrical properties of sensorial units, the interactions between liquids and interfaces have a crucial role. Here, we explore the interaction between dioctadecyldimethylammonium bromide (DODAB) lipid and carbendazim (MBC) pesticide in an e-tongue to discriminate different MBC concentrations in aqueous solutions. The sensorial units were fabricated of gold interdigitated electrodes (IDEs) coated with Layer-by-Layer (LbL) films of DODAB and nickel tetrasulfonatedphthalocyanine (NiTsPc), perylene and 1,2-dipalmitoyl-sn-glycero-3-phospho-(1''-rac-glycerol) (DPPG), namely DODAB/NiTsPc and Perylene/DPPG, respectively. Besides a bare electrode also constituted the e-tongue to distinguish MBC concentrations from 1.0 x 10^{-7} up to 1.0 x 10^{-10} mol L-1, by impedance spectroscopy. The LbL films were manually fabricated obtaining linear growth monitored via UV-Vis absorption spectroscopy. Optical images associated with chemical mapping reveals the presence of small aggregates in the DODAB/NiTsPc LbL film surface. Although the e-tongue was able to discriminate all MBC concentrations by means interactive document map (IDMAP), only the sensorial unit covered with DODAB/NiTsPc LbL film presents satisfactory response. According to the equivalent circuit the main contribution arises from the bulk and film surface due to the interaction between DODAB and MBC indicating selectivity of the sensorial unit. Finally, the adsorption of MBC molecules onto the film surface induced an irreversible process, although there are some frequencies which the sensorial unit response seems to be reversible, as shown by parallel coordinates.

L-Tyrosine Methyl Ester Hydrochloride under high-pressure

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Amino acids N-methylation has been used in synthesis of new peptide-based drugs by pharmaceutical industry [1]. N-methylation has the ability to change hydrogen bonds configuration and molecular conformation of peptide chain, altering their biological functions [1]. Here, we purpose to investigate the vibrational and structural properties of L-Tyrosine Methyl Ester Hydrochloride crystal (LTMEHCl) by high-pressure Raman Spectroscopy. LTMEHCl was grown by slow evaporation method (stirring for 3 h, at 200 rpm and 318 K; pH = 2.15), over a period of 3 months. Powder X-ray diffraction was performed on Empyrean powder diffractometer of the Panalytical (Cu Kα radiation, λ = 1,5418 Å), with 0.02° and 2 s/step in 5-50° 2θ range. High-pressure Raman scattering (1 atm - 9.0 GPa), in a T64000 Jobin Yvon spectrometer, using LTMEHCl and Nujol oil, wasperformed. Laser beam (Ar: Kr laser, k = 514.5 nm, backscattering geometry) was focusedwith Olympus BH40 microscope. LTMEHCl was obtained in monoclinic symmetry. High- pressure Raman results revealed reversible spectral changes in the pressure range of 0.3 -1.3 GPa and 2.8-5.0 GPa, indicating, at least, conformational phase transitions. Notably, methylation increase the flexibility of the LTMEHCl crystal lattice compared to L-tyrosine hydrochloride, which did not exhibit any phase transitions in similar conditions [2].

Acknowledgements:
Authors thank FAPEMA, CNPq and CAPES.

References:

[1] M. Mindt, J.M. Risse, H. Gruß, N. Sewald, B.J. Eikmanns, V.F. Wendisch, , Sci. Rep. vol. 8 (2018).

[2] C.A.A.S.S. dos Santos, J.O. Carvalho, J.G. S. Filho, J.L. Rodrigues, R.J.C. Lima, G.S. Pinheiro, P.T.C. Freire, P.F. F. Filho, Phys. B Condens. Matter. vol. 531 (2018).

Luminescent membranes based on derivative celulose acetate butyrate (CAB) doped with europium complex

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Polymers based on cellulose derivative have been attracting interest considering a sustainable approach. Cellulose Acetate Butyrate (CAB) presentes interesting properties such as film forming, flexibility, high transparence in the visible range. Its solubility in a wide variety of solvents make it useful as an additive in coating compositions. It has been applied as diffuser layer to suppression of the viewing angle dependence on blue OLEDs [1]. Considering these properties CAB arises as potential matrix for immobilization of lanthanides for luminescente applications. In this work, [Eu(tta)3(H2O)2] was incorporated, 0.2 to 3.2% (w:w), in the Eastman CAB-551-0.2 (Butyryl 52wt, Acetyl 2wt and Hydroxyl 1.8% content) Mw 30000 by using ethyl acetate as a solvent. Membranes of CAB-[Eu(tta)3(H2O)2] were synthed by casting and characterized by FTIR-ATR, UV-Vis, TGA and Photoluminescence. Membranes present high transparence in the visible range, flexibility and a broad band in the UV region with maxima at 270 and 340 nm assigned to π-π* excited states of ligand. FTIR-ATR show that complex was well dispersed in the CAB matrix. In addition, the membranes are thermally stable up to approximately 300oC. Excitation spectra monitored at 614 nm (5D0 → 7F2) show a broad band in the UV region with maximum peak shifted to higher energy as a function of complex content. Emission spectra display high intense sharp lines ascribed to 5D0 → 7F0-4 transitions through the antenna affect [2]. CIE diagram show (0.68, 0.32) color coordinates and decay curve, fitted by a single exponential exhibit experimental lifetime of 0.32 ms. The same behavior was observed for all composition studied. suggesting only one Eu3+ local site. The synergism between CAB and complex present potential application for flexible emitting luminescent devices.

Acknowledgments:

FAPESP and INEO (2014/50869-6); CNPq (160631/2022-0)

References:

[1] Organic Electronics 28, 31 and 38, 2016)

[2] J. Lumin. 71 (3), 229-236, 1997

Microstructured design by thermal poling in niobium germanate glasses

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All-optical photonic devices are currently widely investigated as they allow to improve the overall circuit speed which is limited by response time of electronic devices. Advances have being made in this regard, such as the development of optoelectronic devices for integrated circuits [1,2]. This work investigates the structural and non-linear properties of microstructures printed by thermal poling on glass $65GeO_2-20Nb_2O_5-15Na_2O$. Thermal polling was performed using an anode with Pt film deposited with microstructures design forming 40 μm wide squares. The poling system was heated to 265oC for 30 minutes at N_2 atmosphere and a DC bias of 1200 V was applied.

AFM analysis allowed to characterize the morphology of the subanodic surface which presented a depth of about 150 nm. PHASICS data were used to calculate the variation of the refractive index $\Delta n = n(NIC) - n(IC) = -0.085$, where NIC and IC are the regions non in contact and in contact with the conductive electrode. The increase in the index of refraction in the IC region is due to the network polymerization and the higher content of NbO_6 polyhedra. Nonlinear optical analysis were recorded by μ-SHG polarized microscopy and show well localized SHG signals at the edges of the square. This microdevice may besuitable for photonic applications, especially wavelength conversion and electro-optical devices.

Acknowledgements:
The authors would like to thank Brazilian funding agencies FAPESP (2020/01786-1; 2021/11494-0) for financial support.

References
[1] K. Xu, B.Y. Zhang, Y. Hu, M.W. Khan, R. Ou, Q. Ma, C. Shangguan, B. J. Murdoch, W. Chen, X. Wen, G. Ren, J.Z Ou Journal of Materials Chemistry C, 9, 2021, 3115-3121.
[2] H. Chen, C. Wang, Y. Song, J. Tian Nanophotonics, 9:8, 2020, 2107-2124.

Morphological and structural analysis of electrospun graphene oxide and PVDF nanofibers for application in organic optoelectronics.

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In the manufacture of optoelectronics, various commercial techniques are used, such as roll-to-roll printing, screen printing, inkjet printing, foil coating, gravure printing, flexography, spray coating and brush painting. However, each of these techniques has specific disadvantages, such as high temperature heat treatments, application of toxic solvents and disposal of contaminated waste. On the other hand, electrospinning is a promising technique for the production of polymeric fibers on a large scale, with advantages such as versatility, high surface/volume ratio, unidirectional conduction, low cost and environmental sustainability. In addition, electrospinning allows the nanoscale modification of the structural characteristics of optoelectronic components, arousing great interest in the scientific community. Reduced graphene oxide (rGO) is a suitable material for use in flexible conductive electrodes, transparent and stretchable, due to their high conductivity and mechanical resistance, which help in the efficient transport and capture of charges from the active layer to the device. In this job, Poly(vinylidene fluoride) (PVDF) and rGO polymeric fibers were electrospun at different concentrations and characterized by techniques such as optical microscopy, scanning electron microscopy and Fourier transform infrared spectroscopy, with the aim of being used in optoelectronic devices.

Acknowledgements
The authors would like to thank CNPq, LabMEV-FCT/UNESP, INEO and FAPESP. CAPES-PRINT-UNESP 2022 - Unesp Network for the development of organic and flexible electronics - ORGFLEX. This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Reference

[1] GONÇALVES, Hugo Pedra et al. Matéria (Rio de Janeiro), v. 26, 2022.
[2] REDDY, Vundrala Sumedha et al. Polymers, v. 13, n. 21, p. 3746, 2021.
[3] TARCAN, Raluca et al. Journal of Materials Chemistry C, v. 8, n. 4, p. 1198-1224, 2020.

New heterodyne Z-scan technique to measure thethermal and Kerr nonlinearity of materials

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Heterodyne Z-scan technique (HZS) was designed to measure slow nonlinear refraction of materials employing CW laser and dual phase lock-in [1]. In order to do the heterodyne detection, the original setup uses an unusual sinusoidal intensity modulator. Now, by replacing this modulator with a simple optical chopper, we could extend the use of HZS method with ultrashort pulses. In this case, we can measure both the thermal and ultrafast Kerr nonlinear effects. Basically, the dual-phase lock-in detects the pure thermal and the combination of both effects in the Phase and Magnitude signals, respectively. Also, by measuring HZS as a function of chopper repetition rate, it is possible to find the response time of the thermal nonlinearity. Here, we proposed to perform the HZS measurements to discriminate the thermal and Kerr nonlinearity of different materials. We characterized pure CS_2 solvent using ultrafast laser pulses (Carbide, Light Conversion, ~200 fs at 515 nm, 1-1000 kHz repetition rate). This laser is interesting since the thermal lens signal can be controlled by its repetition rate. CS_2 is a well-known material with high Kerr nonlinearity with two-photon absorption thermal lens when high-repetition rate laser is employed. Moreover, linear and circular polarization can discriminate the pure electronic and molecular orientation effects from the effective Kerr nonlinearity [2]. Optical glasses were also characterized.

Acknowledgements:

CNPq and FAPESP.

References:

[1] I. Guedes, L. Misoguti, L. de Boni and S. C. Zilio, J. Appl. Phys 101, 063112 (2007).

[2] M. S. Melhado, T. G. B. de Souza, S. C. Zilio, E. C. Barbano and L. Misoguti, Opt.Express 28, 3352-3360 (2020)

Nonlinear properties and structural rearrangements in thermally poled niobium germanate glasses

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Research on glass materials has gained prominence since adjusting the composition/properties of glasses can result in greater material efficiency. Glasses containing transition metal elements are known to exhibit enhanced luminescent properties of rare-earth ions [1], as well as nonlinear optical (NLO) properties [2]. This work encompasses the structural and NLO characterizations resulting thermal poling in sodium niobium germanate glasses: $(100-x)GeO_2-xNb_2O_5-15Na_2O$ with x = 15, 20 and 25mol%. Thermal poling was also performed for 30 min, at ~265 oC and a DC bias of 1200 V.

A subanionic layer was formed in the region in contact with the conductive homogeneous electrode. Structural rearrangements and nonlinear properties were investigated by micro-Raman and micro-SHG. Sodium depletion and a static electric field implementation occur in the subanionic layer. Structural rearrangements occurs due to decrease in non-binding oxygen (NBO) bonds and O_2 generation due to recombination NBO atoms leading to the release of sodium and oxide ions in the NLO layer. Macro-SHG response was quantified using the Maker fringes technique and the electric field induced second harmonic (EFISH) model was used to simulate the fit and determine the $\chi 2$ susceptibility. The $\chi 2$ values showan increase from 0.42 to 0.76 pm/V with the niobium content.

Acknowledgements:

The authors would like to thank Brazilian funding agencies FAPESP (2020/01786-1; 2021/11494-0) for financial support.

References:

[1] R.R. Gonçalves, Y. Messaddeq, A. Chiasera, Y. Jestin, M. Ferrari, S.J.L Ribeiro, Thin Solid Films, 516, 2008, 3094-3097.

[2] F. Goumeidane, M. Iezid, B. Melik, K. Ouannes, M. Legouera, M. Poulain, T. Satyanarayaran P.S. Prasad, P.V. Rao, Ceramics International, 43, 2017, 11305-11311.

Nonvolatile memory devices with anodized aluminum layer

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Non-volatile metal oxide memory devices are of great interest in the field of information storage. These materials have specific physical properties such as high resistivity and permeability, in addition to transparency over a wide range of wavelengths. In memories, the oxide layer acts as a tunneling barrier between the different layers that make up the device, allowing the storage of electrical charges [1-2]. Film properties depend strongly on the synthesis and/or deposition process. The anodizing is a lower cost alternative, as it does not require a controlled atmosphere. Anodizing is an electrochemical process that involves the controlled oxidation of the metal through a redox reaction, generating a thickening of the oxide layer on the surface of the material. In this work, we performed the deposition of aluminum films on a glass substrate that received the anodizing treatment to grow the native oxide layer. Different voltages and application time and, consequently, current, were used. For electrolyte solution, H_3PO_4 at 0.4mol L^{-1} was used and platinum electrode as cathode. It was observed that increasing the aluminum oxide layer generates an increase in film transparency. The increase in the oxide layer increase transmittance, and consequentlya decrease in the absorbance. We observed dependence between time and voltage appliedin the experiment, with oxide growth being directly proportional. Roughness increases with increasing exposure time. The resultant memory devices with Al/Al_2O_3/MEH-PPV/Al architecture were characterized and showed bipolar behaviour. After electroforming the device show two distinct resistance states: Low Resistance (LRS) and High Resistance States (HRS). Current ratio between LRS and HRS is at least 10^3. Long term retention and endurance cycling are in progress.

Acknowledgements:
CNPq, CAPES, FINEP, FAPESP, UFPI, INEO References

References:
[1] Kim, J. J., Advanced Materials 23, (2011).
[2] Cho, B., Appl Phys Lett 97, (2010).

One-pot synthesis of pinacolone derivatives from acetophenones

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During the investigation to synthe 3,7-di-t-butylphenothiazine (a potential good π-electron donor for photoinduced charge separation process) from phenothiazine by using zinc and tert-butyl chloride as reagents,[1] we observed that these conditions led to the formation of 3,3-Diphenyl-2-butanone (a pinacolone derivative) in a one-pot, single step fashion, starting from commercial acetophenone. According to the literature, the synthesis of pinacolone derivatives directly from acetophenones is not well reported. To assess the scope of the methodology, different acetophenones were tested and the corresponding pinacolones were obtained in moderate yields. Structure elucidation was supported by ^{1}H, ^{13}C NMR spectra and GC-MS (gas chromatography-mass spectrometry) and showed a high degree of purity. This methodology can be useful in materials science since some pinacolone derivatives have applications as radical initiators in polymer synthesis.[2]

References:
[1] Paul, S.; Nanda, P.; Gupta, R.; Loupy, A. Zinc Mediated Friedel-Crafts Acylation in Solvent-Free Conditions under Microwave Irradiation. SYNTHESIS 2003, 18, 2877-2881.
[2] Shinji Watanabe, S.; Murayama,H.; Murata, M.; Masuda, Y.; Tanabe, M.; Imai, Y. Synthesis and Characterization of New Aromatic Polyestersand a Polyether Derived from 2,2-Bis(4-Hydroxyphenyl)-1,2-Diphenylethanone Journal of Polymer Science: Part A: Polymer Chemistry 1998, 36, 2229 -2235.

Organic solar cells based on Poly(3-hexylthiophene), PCBM and graphene

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Environmental concerns reinforce the relevance of clean and viable renewable energy sources. Due to its abundant energy resource available and lower production costs, solar energy is one of the best options. Organic photovoltaic devices (OPVs) are made of organic (carbon-based) semiconducting small molecules or polymers for converting incident sunlight into electrical power [1]. Photovoltaic cells generate electricity directly via the photovoltaic effect. In this work, the OPVs were built in the FTO/PEDOT:PSS/P3HT/Al, FTO/PEDOT:PSS:GO/P3HT/Al and FTO/PEDOT:PSS/P3HT:PCBM/Al device architecture. We performed current-voltage measurements in temperatures ranging from 80 to 300 K. We also carried out on the parameters that define its electrical properties, such as the lifetime of photogenerated carriers and its electronic mobility.

Acknowledgements:
The authors thank the following Brazilian Development Agencies: CNPq, INCT/INEO (Brazil), FAPEMAT, PROPeq/UFMT and CTI Renato Archer.

References:
[1] Głowacki, E.D., Sariciftci, N.S., Tang, C.W., Encyclopedia of Sustainability Science and Technology, 7553-7584 (2012)

Photophysical and electrochemical properties of three novel fluorescent Benzazole derivatives and their interaction with fullerenes

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Here, we present the study of the photophysical and electrochemical properties of three novel fluorescent Benzazole derivatives, constituted by a central sulfur heteroatom, or oxygen or nitrogen and their interaction with fullerenes in solution. The photophysical properties were studied in liquid (solution) and solid (film) state at room temperature using absorption, stationary fluorescence and time-resolved fluorescence spectroscopy. The molecules exhibited absorption in the visible region, high fluorescence quantum efficiencies, and monoexponential (a single excited state lifetime) fluorescent decays. The energy values of the molecular orbitals (HOMO, LUMO) of the molecules were estimated using voltammetry (VC). The fluorescence intensities of the compounds in toluene solution with different concentrations of fullerenes C60 and derivative Pyrrolidine tris-acid ethyl ester (PyC60) were quenched by simultaneous mechanisms (dynamic and static) of fluorescence quenching. The fluorescence suppression constant (Ksv) and the radio of the effective suppression spheres were obtained from Stern-Volmer plots. The three novel fluorescent Benzazole derivatives presented optoelectronic characteristics suitable for their application in organic electronic devices, specially for solar energy conversion.

Acknowledgements: CNPq, FAPEMIG

Photophysical Characterization of 2,1,3-benzoxadiazole: Building Blocks for Optoelectronic Devices

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Benzochalcogendiazole heterocycles have been identified as potential building blocks for the design and development of optoelectronic devices such as organic light-emitting diodes (OLEDs) and solar cells. These heterocycles also exhibit promising applications in nonlinear optics as well as in various chemical transformations such as oxidation, reduction, and coupling reactions. They can also function as fluorescent probes for the detection of heavy metal ions, pH, and biological molecules, and as photocatalysts. In this study, we characterized the photophysical properties of derivatives of 2,1,3-benzoxadiazole, in which the structure of the central nucleus promotes the formation of highly organized molecular arrangements characteristic of liquid crystals. Specifically, the photophysical properties of 2,1,3-benzoxadiazole compounds with different symmetric branches, 4b ($C_7H_{16}0$) and 4e ($C_{12}H_{25}NO_3$), were studied in solvents with different polarities and in film form. The absorbance of both compounds occurs in the visible region, with a peak at 413-420 nm for 4e and slightly higher at 430-442 nm for 4b. The fluorescence quantum yield varied from 0.3 to 0.8, depending on the solvent, and the Stokes shift ranged from 2300 to 4500 cm-1 for both solvents. The excited state lifetimes were approximately 2.0 ns. The photophysical properties of the films were similar to those of the solution samples in terms of maximum absorption and the shape of the absorption and fluorescence spectra. However, the Stokes shift of the films was higher. The values of the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) were estimated for compounds 4b and 4e. The estimated values for HOMO and LUMO were -5.00 eV and -2.23 eV, respectively, for compound 4b, and -5.01 eV and -2.06 eV, respectively, for compound 4e. The estimated values for HOMO and LUMO suggest that these compounds are promising candidates for use in OLEDs, organic solar cells, and

Polarization-resolved Z-scan technique to discriminate the nonlinear refractive index of optical glasses

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Synthesis of new optical glasses with high nonlinear refractive index plays an important role in optics and photonics, especially for all-optical switch applications. However, depending on laser pulse width, wavelength, and repetition rate, different nonlinear refractive effects can arise simultaneously. Indeed, there are three main nonlinear effects that can emerge in materials: nonresonant electronic, molecular orientational and thermal, which need to be discriminated. For example, pump and probe techniques can be employed [1] to discriminate these effects based on their response times, but they require complex two or more beam setup. On the other hand, we can use a single-beam method: the polarization-resolved Z-scan (PRZS) [2], to discriminate them. Our technique explores the laser polarization (linear and circular) and repetition rate to do so. This was possible thanks to newly available femtosecond pulses systems which repetition rate can be controlled from very high to low where thermal effect takes place or not, respectively, such as the Carbide laser from Light Conversion (515 nm, ~200 fs, with controllable pulse repetition rate:1-1000 kHz). Here, we propose using PRZS to characterize optical glasses. To validate our methodology, we conducted measurements on three significant glasses: SF6, LaSF-N30, and a neutral density filter. Our findings revealed the presence of pure electronic and thermal effects, while no orientational contribution was observed.

Acknowledgements:
CNPq, FAPESP.

References:

[1] M. Reichert, H. Hu, M.R. Ferdinandus, M. Seidel, P. Zhao, T. R. Ensley, … and E. W. Van Stryland, Optica 1(6), 436-445 (2014).
[2] R. M. Moysés, E. C. Barbano and L. Misoguti, JOSA B 40(4), C60-C66 (2023).

Preparation and characterization of cesium carbonatedoped indium tin oxide for low work function electrode

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Clean energy has become a topic of ever increasing interest in recent times. An effective method of producing clean energy is through Organic solar cells (OSCs)[1]. A crucial mechanism in this device is the donor-acceptor (D-A) heterojunction, where photoinduced charge separation occurs (electrons and holes). Commonly, Indium Tin Oxide (ITO) is used as anode due to its high work function (4.62-4.81 eV)[2] and a metal with low work functionis used as anode. However, these low work function metals are easily oxidized, making theOSCs less durable in ambient conditions. By preparing inverted cells, this problem is circumvented. In this cells, ITOs work function is lowered by chemical modification and so it can play the role of cathode and a high work function metal can work as anode. It has been noted that by doping its surface with cesium carbonate ($Cs2CO3$) its work function is lowered to about 2.5 eV [3]. However, it is not clear how reproducible is the relation between chemical modification and work function lowering and the mechanism of such effect. In this work we prepared and characterized pellets of $Cs2CO3$ doped ITO nanoparticles by ball milling and thermal annealing. We determine the distribution and quantify the $Cs2CO3$ on the ITO using Energy dispersive spectroscopy (EDS). Atomic Force Microscope is used to analyse the topology of the pellets. X-ray photoelectron spectroscopy (XPS) is used to measure the work function of the materials.

Acknowledgments:

We acknowledge FAPESP for financial support.

References

[1] G. Yu, J. Gao, J. C. Hummelen, F. Wudl, and A. J. Heeger, Science 270, 1789 (1995).

[2] S. D. Nehate, A. Prakash, P. D. Mani, and K. B. Sundaram, ECS Journal of Solid StateScience and Technology 7, P87 (2018)

[3] M.-H. Chen and C.-I. Wu, Journal of Applied Physics 104, 113713 (2008).

Preparation and characterization of phenothiazine oligomers as low bandgap organic system

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Donor-acceptor molecular systems for organic photovoltaics (OPVs) are composed by an association of an low-bandgap oligomer with an electron defficient acceptor.[1] Thus, the design and preparation of low-bandgap oligomers with high absorbance in the solar spectrum and high hole conductivity is an important step towards efficient OPVs. We hereby propose an association of oligophenothiazines that can be formed of benzoid and quinoid structures to prepare such low-bandgap oligomers. The benzoid units are electron donors while the quinoid are acceptors, thus, benzoid-quinoid association is a smart strategy to prepare intramolecular "pull-push" electron conjugations that favors the bandgap lowering. However, it is not clear how the molecular size and the ratio between quinoid and benzoid structures inpact the bandgap of the oligomer, neither it is well established chemical routes to obtain oligomers with controlled molecular structure. In a first approach, we carried out an interfacial oxidative polymerization (IOP) of the monomer using ammonium persulfate (APS) as the oxidizing agent in a toluene-isopropanol mixture[2]. We also prepared oligomers by the IOP method and APS oxidizer using glacial acetic acid solvent and higher temperature[3]. The quinoid/benzoid ratios and cross linking characteristics were determined by liquid-state nuclear magnetic resonance (NMR) spectroscopy. In addition, we report the optical characterization from UV-VIS absorption and the optical gap of the oligomers.

Acknowledgements:
FAPESP, CCNH-UFABC

References:

[1] Eva Bundgaard and Frederik C. Krebs. Low band gap polymers for organic photovoltaics. Sol. Energ. Mat. Sol. C., 91(11):954-985, 2007.
[2] S. Zh. Ozkan, G. N. Bondarenko, A. V. Orlov, and G. P. Karpacheva. Interfacial oxidative polymerization of phenothiazine. Polymer Science Series B, 51(5):149-156, Jun2009.
[3]X. Wang, G. Li, Y. Han, F. Wang, J. Chu, T. Cai, B. Wang, Z. Song, ChemSusChem2021, 14, 3174.

Preparation of stable luminescent films containing chitosan and neutral conjugated polymer as a compatible matrix for small molecules

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Conjugated polymers embedded in biopolymer films are promising materials due to their luminescent properties and the possibility to apply in photoresponsive devices [1]. Chitosan is a biocompatible material with the ability to form self-standing and malleable films [2]. Even though chitosan was shown to be miscible and compatible with other biopolymers andionic polymers, the incorporation of insoluble neutral conjugated polymers and small molecules could be challenging. The objective of this work is to understand how a conjugated polymer, poly(3-hexylthiophene-co-1,4-phenylene) (PTPh), can be incorporated into a self-supporting film containing chitosan and how this new film can enhance thedispersion of small molecules. The 4-aminoazobenzene (4-AAB) was chosen as a small molecule since its cis-trans photoisomerization provides information on the localenvironment in the microheterogeneous film. Using spectroscopy and microscopy it was observed the complete phase separation of 4-AAB into chitosan films. However, in presenceof PTPh, compatible and stable films were obtained. PTPh luminescence showed to changein presence of 4-AAB. Thus, the luminescent properties of PTPh when effectively dispersedin a chitosan matrix can be used to gain information on the reasons for the much betterdispersion of 4-AAB into the chitosan matrix. These results are a consequence of the molecular interactions between all components in the film. Hence, this work provides fundamental and methodological approaches to incorporating small molecules into stable, self-standing, and luminescent polymeric films for potential applications in fluorescencemodulation, sensors, and optoelectronic devices.

Acknowledgements:
CNPq (401109/2014-3), Capes, Fapesp (2015/14681-5, 2018/23524-9, 2019/15498-0).

References:
[1] Pinto, R J B, et al. J Appl Polym, v 131, p 41169/1-41169/21, 2014.
[2] Rinaudo, M. Prog Polym Sci, v 31, p 603-632, 2006.

Resistive memory degradation based on semiconductor organic polymer

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The morphological characteristics and alterations of the active layers and electrodes due to the degradation of the resistive memories after several write/read/erase/read cycles need to be analyzed. In this work, we focus on the degradation of a memory device formed by poly (2-methoxy-5-(2''ethylhexyloxy)-1,4-phenylene vinylene) (MEH-PPV) as an active layer sandwiched between two electrodes, Indium Tin Oxide (ITO) and Aluminum (Al). It was observed that the aluminum electrode after more than 100 cycles began to show regions with defects. The Raman and Scanning Electron Microscopy (SEM) techniques together with EDS were used to understand the structural and conformational changes due to device degradation. The SEM image of the aluminum electrode showed regions of permanent degradation (holes). The EDS analysis shows that in this region there are remnants of materials that formed the device: In the center ITO signal, and in the border region mix signals of aluminum, polymer, and ITO. All layers are altered, including the ITO electrode. The Confocal Raman microscopy shows different characteristic signals: MEH-PPV in the border of the hole, ITO, and glass in the center. Similarly, Li et. al. [2] analyzed the MEH-PPV/aluminum interface obtaining practically the same features. Thus, in our case, spectral characteristics indicate weak MEH-PPV/Al interfacial interaction, therefore the formation ofa native aluminum oxide layer at the interface may be considered since Al has a strong affinity with oxygen. It is possible that the degradation of the device is the result of several processes such as a strong discharge process and the production of O2 gas when voltage was applied repeatedly to make the read-write-read-erase cycles.

Acknowledgments:

CNPq, CAPES, FINEP, FAPESP, UFPI, INEO

References:

[1] MIAO, Feng, et. al. Advanced Materials, v. 23, n. 47, p. 5633-5640, 2011.

[2] LI, Dongbo; et al. Applied Physics Letters, v. 100, n. 14, p. 141907, 2012.

REVEALING A GIANT MOLECULAR SECOND HARMONIC GENERATION IN DONOR-EMBEDDED POLYBINAPHTHALENES BY FEMTOSECOND TUNABLE HRS TECHNIQUE

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The high demand for new materials with enhanced optical features has led to a growing interest in molecular nonlinear optics. This area of research has facilitate the creation of more efficient materials and a decrease in the cost of recent technologies. In particular, the macromolecules has received a special attention due your high symmetry breaking being a excellent candidate for presents a great values of second order optical susceptibility [1] $\chi 2$. The polybinaphthalenes has a ideal macromolecular structure to link differents chromophores to optimize the nonlinear optical responses [2]. In this context, we determined the spectral dependence of the first order molecular hyperpolarizability $\beta(\lambda)$ of four donor-embedded polybinaphthalenes (DEP) structures in solution. For this, we apply the tunable femtosecond Hyper Rayleigh Scattering (HRS) technique [3]. The results showhigh β values for all studied macromolecules, with peak values ranging from 2500 to 16000 ($x10^{-30}$ cm^4 $statV^{-1}$). Concerning the values of β for pNA molecule, the (DEP) are c.a. 25 to 160times higher at the enhancement resonance region. This giant molecular second harmonic generation makes these class of macromolecules an exceptional candidates for photonic devices, such as, in the fabrication of highly efficient organic crystals for second harmonic generation.

References:

[1] Koeckelberghs G, Sioncke S, Verbiest T, et al.. Macromolecules. 2003;36(26):9736-9741.

[2] Koeckelberghs G, De Groof L, Picard I, Verbiest T, Persoons A, Samyn C. Macromolecular Rapid Communications. 2005;26(11):905-910.

[3] Clays K, Persoons A. Physical Review Letters. 1991;66(23):2980-2983.

Solvatochromism of Eu^{3+} complex based on 3,5-Dihydroxybenzoic acid and 2-hydroxy-4-methoxy-benzophenone.

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The design of lanthanide complexes has been an important research goal owing to their applications such as lighting sources and sensors. This study describes the synthesis, characterization, and solvatochromic properties of a Eu^{3+} complex, using cheaper ligands as antennas when compared to usual ones, obtained as follows: $Eu(NO_3)_{3(aq)}$ was added to a solution of 3,5-Dihydroxybenzoic acid, Hdic, and 2-hydroxy-4-methoxy-benzophenone, Hbzf, (ratio 1:2) in DMF, and the precipitate was isolated after filtration and washing with a mixture of water/DMF. FTIR showed the coordination of Eu^{3+} via carbonyl group due to the shift of 42 cm^{-1} in the ν_{as}(C=O) stretching, and Eu^{3+} titration, as well as CHN analysis results, allowed the proposition of the stochiometric formula $[Eu(dic)_2(bzf)(DMF)(H_2O)]$. The produced complex exhibits the characteristics *f-f* Eu^{3+} transitions [1] and τ = 0.20 ms. The T_1 was calculated via an isostructural Gd^{3+} complex (19.710 cm^{-1}), suggesting that the ligands act as a suitable antenna for Eu^{3+}. In addition, a solvatochromic study was performed using several solvents with different polarities to explore the spectroscopic properties of the complex. The solvent sensitivity to the complex was evaluated by the Lippert-Mataga plot, where the polarity function exhibits a good correlation with pseudo-Stoke's shift (R^2 = 0.94), when compared to the literature [2]. Finally, the observed correlation suggests that the solvatochromic shift can be explained by a dielectric solute-solvent interaction. In conclusion, the desired complex was successfully synthed and has promising optical features in sensing based on its solvatochromic properties.

Acknowledgements:

CAPES, CNPq, LAMAQ, IQ Unesp, DAQBi-UTFPR and UFSC.

References:

[1] K. Binnemans, Coordination Chemistry Reviews, v. 295, p. 1-45, (2015).

[2] J. Solanki and K. Surati, Materials Today: Proceedings, v. 29, p. 1006-1012, (2020).

Structural and Vibrational properties of Glycinium Maleate single crystal under high-temperature conditions

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Physical properties of organic salts can change under extreme thermodynamics conditions [1-2]. Here, we investigated the vibrational and structural properties of glycinium maleate crystal (GM) via High-Temperature Raman Spectroscopy (HTRS) and High-Temperature Powder X-ray diffraction (HTPXRD). GM was obtained in 38 days by slow evaporation method from solutions of glycine and maleic acid (Sigma Aldrich, 99%) at 1:1 ratio. HTPXRD were performed on Empyrean powder diffractometer of Panalytical (Cu Kα radiation, λ = 1,5418 Å); with 0.02° and 2s/ste 2θ range 5-50°. GM was studied by Thermogravimetricand Differential Thermal Analysis (TG-DTA) using a DTG-60 instrument (Shimadzu) - Al crucible, N2 atmosphere (flux of 100 mL/min.), heating rate of 5 K/min. HTRS, from 298 to 438 K, was performed on TriVista spectrometer (Princeton Instruments) and He-Ne laser (λ = 632.8 nm). HTPXRD suggested conformational modifications in the range of 403-423 K, owing to intensity inversion of peaks at 24,04° and 26,88°. TG-DTA curves indicated good thermal stability up to 397 K and temperatures of melting process, thermal decomposition, and glycine liberation at 415, 438 e 499 K. Agreeing with that, HTRS reveal spectral changes around 408 K due to modifications in bands associated with glycine atomic groups, followed by a phase transformation caused likely by glycine liberation.

Acknowledgements:

Authors thank CNPq and CAPES.

References:

[1] C.A.A.S. Santos, R.J.C. Lima, P.F.F. Filho, A.O. do. Santos, J.A. Lima, P.T.C. Freire, J. Phys. Chem. Solids. vol.136, 109129 (2020).

[2] N. Singh, B.K. Singh, M.K. Gupta, B.Kumar, Optics Communications vol. 285, 659-664 (2012).

Study of a perylene diimide derivative liquid crystal for organic solar cells applications

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Organic solar cells (OSCs) are an interesting option for reducing environmental impact. The main benefit is the ability to produce lightweight, flexible and portable devices that can be used in daily life. In this field, the search for non-fullerene acceptor materials (NFAs) is currently one of the main focuses. Columnar liquid crystals (ColLCs) based on perylene diimide are among the promising materials to replace fullerenes. ColLCs have the advantage that molecular alignment can be controlled to obtain charge transport in the desired direction [1]. By combining their excellent optical, self-assembly and semiconducting properties, ColLCs became very attractive for applications in OSCs. In this work, the polymer PFO-DBT was used as the donor (D), and a room-temperature ColLC based on a benzoperylene diimide diester as the electron acceptor (A). The absorptionspectra of the two materials are complementary, covering a range from 300 to 650 nm. Inthe presence of the polymer, the photoluminescence (PL) of the ColLC is quenched. This quenching can be caused by a D:A charge transfer or even by an energy transfer between the materials. Thin films of the mixtures were aligned with the ColLC molecules in face-on orientation. Higher absorption and PL intensities were obtained from aligned samples. AFM measurements revealed homogeneous surfaces with a reduction in the roughness values for the mixture films. Also, different amounts of the solvent additive DIO were added to the polymer:ColLC films. The crystallinity of the films increased by increasing DIO concentration, indicating an improved morphology. Finally, electrical measurements were carried out to obtain the performance of the ColLC as an acceptor material and also as the electron transport layer.

Acknowledgements:
CAPES, CAPES-COFECUB Project, CNPq, and INCT/INEO

References:

[1] J. Eccher, G. C. Faria, H. Bock, H. von Seggern and I. H. Bechtold, 5, 11935-11943 (2013)

Study of composites involving PEDOT:PSS and CuNPsin anticorrosive coating of CA-50 carbon steel used in reinforced concrete structures.

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Corrosion is a common phenomenon in reinforced concrete structures. This phenomenon, however, results in a significant loss of resistance to tensile stresses in structures, making them more fragile and inefficient for various applications[1]. In order to avoid and/or reduce this pathological manifestation, the use of composite materials involving conjugated polymers and metallic nanoparticles for protecting reinforced concrete structures has been studied[2]. Thus, this work studied the formation of a composite for covering CA-50 steel reinforcements, containing epoxy (commercial anticorrosive paint), poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) (PEDOT:PSS) polymer, very popular and with good corrosion inhibition characteristics[3], and copper nanoparticles (CuNPs) which can aid in adhesion to the substrate and protect against microbial corrosion. Finally, the corrosion parameters from the CA50 steel were analyzed using the potentiodynamic polarization technique. For this, four solutions that simulated pore waters existing inside the reinforced concrete were used, namely: AP1 Ca(OH)2 sat. + 0,004% NaOH + 0,1% KOH; AP2 Ca(OH)2 sat. + 0,004% NaOH + 0,1% KOH + 1,0% NaCl; AP3 0,20% NaOH + 0,20% KOH e AP4 0,004% NaOH + 0,1% KOH + 1,0% NaCl. Through the aforementioned test, it was observed that AP4 was the most aggressive pore water for this steel. Scanning electron microscopy (SEM) of the steel without the coating showed the formation of pores after carrying out the linear polarization with the AP4 solution, and showed that the resulting corrosion product was essentially iron oxide.

Acknowledgments:
We gratefully acknowledge CAPES, INEO, FAPESP(2014/50869-6), CNPq(310647/2020-7).

References:
[1] GENTIL, V. Corrosão, 7th ed. RJ: Ed. LTC, 2021.
[2] SOARES, V. P. Revestimentos poliméricos condutores à base de resina epóxi. p. 1-76, 2018.
[3] HOU, J. Anticorrosion performance of epoxy coatings containing small amount of inherently conducting PEDOT:PSS. Technol, v.29, 2013.

Study of energy transfer in self-assembled structures of dipeptides containing carbazole derivatives via Fluorescence Lifetime Imaging Microscopy.

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The first publications of self-organizing structures of organic molecules appeared in 1976[1]. However, only in 1993, Ghadiri et al, published a work about the synthesis and characterization of peptide nanotubes (PNT)[2]. In addition to nanotubes, peptides are reported in the literature to organize themselves in various structures such as nanovesicles nanowires, hydrogels, nanofibers, and others[3-4]. Studies indicate that the interactions that control the formation of these self-organizing structures may become more dominant as the solvent and concentration solution varies, and consequently the effects on the self-organization of the structures. In this work self-organizing structures of two dipeptites: Phe-Phe and Tyr-Phe, were produced using polar and nonpolar solvents: deionized water, methanol and tetrahydrofuran and adding carbazole derivatives in the samples. The formed structures have fluorescence emission in the blue and green region. Fluorescence lifetime imaging microscopy (FLIM) was used to image the structures formed in order to obtain the fluorescence lifetime parameters of the structures for the dynamic study of the energy transfer processes that occur in these systems. It was observed that the dynamics of the processes change significantly between the structures formed and between the dipeptides used. These processes establish information of extreme importance for the application of these systems in optical sensors, photoluminescent devices and the active layer of photovoltaic systems.

Acknowledgments: CAPES, CNPq and FAPEG for the financial support.

References:

[1] Lotz, B, et al. J. Mol. Biol. 106, 915-942 (1976).
[2] Ghadiri, M. R, et al. Nature 366, 324-327 (1993)
[3] Acet, Omur, et al. Colloids and Surfaces B: Biointerfaces, 113031 (2022).
[4] Zhu, P, et al. Chem. A Eur. J. 16, 3176-3183 (2010).

Study of photophysical and nonlinear optical properties of new non-symmetric 1,3,4-oxadiazole derivatives.

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In recent years, many research groups have carried out theoretical and experimental investigations on nonlinear optical (NLO) organic chromophores [1] for applications in electronics and photonics [2]. In this work we performed the computational modeling of 15 chromophores, with 14 of them having novel structures and studied their electronic properties. Among these, we synthed and characterized 10 chromophores containing the heterocyclic 1,3,4-oxadiazole as the core of the molecule structure. The center of the structure was attached on one side to phenylene and alkyl end groups, and on the other side to thiophene or pyridine groups. We analyzed the effects of varying the terminal group and the aromatic bridge (oxadiazole + phenylene group) on the nonlinear optical properties (NLO) of chromophores. We found that variation in the terminal group has a greater influence on the NLO properties than a change in the central part of the molecule [3]. We investigated the photophysical and nonlinear optical properties by studying luminescence and hyper Rayleigh scattering (HRS) respectively. The computational study of the nonlinear optical properties was performed through density functional theory (DFT-TD) using the B3LYP functional and the 6-31+G(d,p) basis set. The compounds showed high values of hyperpolarizabilities, indicating that they can be applied in optoelectronic devices.

Acknowledgments:
Capes, FACEP, CNPq

References:
[1] Amalanathan M, Hubert JI, Rastogi VK, Spectrochimica Acta A 108., 256 (2013)
[2] Badshah A, Nazar MF, Mahmood A, Ahmed W, Abdullah MI, Zafar, MN, Rana UA, Journal of Molecular Structure 103., 1071 (2014)
[3] Oliveira MSS, Santos ABS, Ferraz, TVB, Moura GLC, Falcão EHL, Chemical Physics Impact 6., 100102 (2023)

Study of the spectroscopic properties of perylene in differents solvents

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Abstract: Organic molecules have been the target of several studies for developing applications in Optics and Photonics [1]. Specifically, these molecules are widely used in the nonlinear optical regime, when the intensity of the excitation light is comparable with the interatomic electromagnetic field. Some nonlinear processes, such as microfabrication, excited fluorescence microscopy and optical storage, allow technological advancement in several areas. In this project, we aim to study the nonlinear effects of the pure commercial perylene, a conjugated organic molecule, specifically the absorption of two photons, in three different solutions, they being the toluol, the dichloromethane and the chloroform. Perylene and its derivatives are already used in applications, such as in optoelectronic devices, in dye lasers and gas sensors, due to their high fluorescence. Among the available techniques to study optical nonlinearities, the Z-Scan[2] stands out. This technique consists, in general terms, of varying the position of the sample along the beam focusing axis, analyzing the normalized transmittance. In addition, measurements of linear parameters, such as molar absorptivity, fluorescence quantum efficiency, fluorescence lifetime, anisotropy coefficient and dipole moment difference between ground state and excited state, determined by linear spectroscopic techniques, were performed. We observed interesting linear optical processes, as well as high two-photon absorption cross-section for perylene, prompting this compound for application in Photonics.

Acknowledgements:

FAPESP, Photonics Group and André Romero

References:

[1] PRASAD, P. N. ; WILLIANS, D. J. New York: John Wiley Sons. , 1991. v. 1. p. 320.

[2] SHEIK-BAHAE, M. ; SAID, A. A. ; WEI, T.-H. ; HAGAN, D. J. ; STRYLAND, E. W. V. IEEE Journal of Quantum Electronics, v. 26, n. 4, p. 760-769, 1990.

Synthesis and characterization of glycine crystal complexed with copper (II) (*Cis*-[Cu(Gly)$_2$].H$_2$O)

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Copper(II) (Cu(II)) has the ability to accept electrons from ligands, resulting in charge transfer and complex formation [1]. Therefore, these study aimed to synthesize and characterize crystals of glycine complexed with Cu(II) through slow solvent evaporation. A 2:1 molar ratio solution of glycine and $CuSO_4.5H_2O$ in deionized water, was stirred for 2 hours at room temperature (~299 K), and then adjusted to pH 7.0 with NaOH. The solution was left at rest on a bench at a temperature of ~299 K for crystal growth. The material was characterized by refined XRD using the Rietveld method, FTIR, TGA-DTA, and DSC. Blue needle-like crystals were obtained in 7 days, crystallizing in an orthorhombic system with space group P212121 with four asymmetric units per unit cell (Z = 4), consistent with literature data [2]. Bands identified in the FTIR spectrum showed C–N bonds in the range of 1250 to 1020 cm^{-1}, and characteristic bands of NH^{3+}, C = 0, CH_2 groups. Through TGA-DTA and DSC, endothermic events, dehydration, and fusion were observed. From the results, glycine acted as a bidentate ligand and formed a complex with Cu(II), with potential in biological and optical applications.

Acknowledgements:
We are thankful the CAPES, CNPQ, FAPEMA, PPGCM-UFMA and LER.

References:

[1] R. M. Williams et al., "Photophysical, electrochemical and electrochromic properties of copper-bis(4,4'-dimethyl-6,6'-diphenyl-2,2'-bipyridine) complexes," Coord. Chem. Rev., pp.253-261, 2002.

[2] S. Konar et al., "Structural determination and characterization of copper and zinc bis-glycinates with X-ray crystallography and mass spectrometry," J Coord Chem, vol. 63, no. 19, pp. 3335-3347.

Synthesis and characterization of silver nanowires and reduced graphene oxide rich in hydroxyl groups as electrodes materials for flexible organic electronics applications

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Transparent Conductive Electrodes (TCEs) have been widely studied due to the growing need to produce new Organic Devices. One of the most widely used TCEs is Indium Tin Oxide (ITO - Sn:In_2O_3), which is mainly used as a transparent conductive thin film in electronic devices. However, ITO is rigid, brittle, and has high commercial costs, which makes it unsuitable for flexible electronic devices. Due to these limitations, several carbon and metallic nanomaterials have been used for the development of electrodes with high transparency and conductivity, including Graphene Oxide (GO) and Silver Nanowires (AgNWs). In this work, the synthesis and characterization of Silver Nanowires and reduced Hydroxyl-rich Graphene Oxide (rHGO) were carried out aiming to be used as efficient TCEs for Flexible Organic Devices. The synthesis of AgNWs was performed using a modified polyol method [1], while the synthesis of rHGO was prepared using a chemical route based on the modified Hummers method [2]. These two nanomaterials were used to produce and optimize hybrid films of rHGO and AgNWs, which combine the excellent adhesion and flexibility of GO with the high conductivity and transparency, and the low sheet resistance of AgNWs. Samples of these nanomaterials were analyzed using different spectroscopic techniques and electrical characterizations. Optimized hybrid films showed sheet resistances ranging from 10.3 Ohm/sq to 32.2 Ohm/sq with transmittances between 82.6% and 90.1%. These results show that our TCEs are comparable to the ITO films, which have sheet resistance between 10 Ohm/sq and 30 Ohm/sq and transmittance above 90%, with the advantage of being suitable for flexible organic electronics applications.

Acknowledgments:
CNPq, CAPES and FAPEMIG supported this work

References:

[1] VASEEM, Mohammad et al., Flexible and Printed Electronics, v. 7, n. 4, p. 044001 (2022)
[2] LIMA, A. H. et al., The European Physical Journal B, v. 93, n. 6, p. 105 (2020)

Synthesis and optical, luminescent, and structural characterization of Er^{3+}-doped germane-tellurite glasses

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Germane-tellurite glasses doped with Er^{3+} were fabricated by conventional melt-quenching method with the chemical composition: (60-x)% TeO_2- 20% ZnO - 10% GeO_2- 10% Na_2O - x% Er_2O_3, where x represents the changes in the %mol concentration of Er_2O_3 in the samples: (x = 0.00, 0.001, 0.01, 0.05 and 0.1). The structural properties were studied by X-ray diffraction, Raman and FTIR [1]. The spectroscopic properties in the visible and near-infrared regions are studied by means UV-Vis-NIR absorption, refractive index, lifetime, emission, excitation, and photoluminescence measurements and afterward, the influence of Er^{3+} doping on the vitreous structure was studied, analyzing the optical bandgaps, Urbach energies, Judd-Ofelt parameters, and boson peaks [1]. The optical properties of the glasses indicate good transparency in the visible region, as well as a dependence between the content of Er^{3+} doping and the hypersensitive band of Er^{3+} ($^2H_{11/2}\rightarrow{}^4I_{15/2}$) and also the energy transfer mechanisms in the up-conversion process of glasses are described [2]. These results provide a complete explanation of the dynamics of Er^{3+} ions within germane-tellurite glasses for laser and photonic applications [3].

Acknowledgments: Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) - Finance Code 001, the São Paulo Research Foundation (FAPESP) under the grant 2013/07276-1, and Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) through the Centro de Pesquisas em Óptica e Fotônica (CePOF)-São Paulo-Brazil (process 314505/2021-0).

References:

[1] J.L. Clabel H et al., Phy. Chem. Chem. Phy. 22, 15022 (2020)
[2] V.A.G. Rivera et al., Opt. Mat. 6, 888 (2011)
[3] G. Lozano et al., J. Alls. Comp 948, 1 (2023)

Systematic Characterization of Cumarin 6 in Different Solvents and Concentrations for Photophysical Analysis and Technological Applications

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Coumarin 6 has been used as a spectroscopic standard due to its high quantum efficiency of singlet state formation. In this study, a systematic characterization of Coumarin 6 was performed in different solvents and concentrations by calculating the fluorescence quantum efficiency using the comparative method and the excited state lifetimes. The use of Coumarin 6 as a spectroscopic standard was found to be suitable at concentrations between 0.735x10^{-06} and 2.20x10 $^{-06}$mol/L, as there were no changes in the shape of the optical absorption and fluorescence spectra. Within this concentration range, there was no significant variation in the efficiency or lifetime of the excited state. At the concentrations used, the polarity of the solvent was found to be the most important factor, as the Stokes shift as a function of solvent polarity (Lippert-Mataga diagram) exhibited a linear behavior even for protic solvents. There was no formation of aggregates or hydrogen bonds at the concentrations and solvents used. In different solvents, higher quantum efficiency of fluorescence was observed for more polar solvents. The excited state lifetimes of Coumarin6 were found to be around 2.3 to 2.6 ns for different solvents, with nonradiative constants being predominant in methanol and acetonitrile, and radiative constants being predominant in chloroform and dichloromethane. Knowledge of the photophysical parameters of reference standards is extremely important for calculating the photophysical properties of new compounds. In addition, controlling the photophysical processes of any chromophore promotes the design of technological applications for organic chromophores. Chromophores such as Coumarin 6 also have applications as fluorescent probes for investigating the polarity of the environment, the efficiency of chemical reactions, tracking of nanodrugs, fabrication of active layers of solar cells, and many other photonic processes.

Systematic study of the $Cu_{2-x}Se$ electrode and it'sinfluence on charge transport mechanisms

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The field of organic semiconductor electronic devices has created a need to find new materials, due to it's advancement, to replace the traditional indium-doped tin oxide (ITO) and fluorine-doped tin oxide (FTO) electrodes. This work was a continuation of the research developed by Silva et al.[1]. Here, a study was made of the change in the thickness of the $Cu_{2-x}Se$ electrode during it's chemical production process. The $Cu_{2-x}Se$ electrode was selected due to it's electrical, optical and mechanical properties presented in the literature. The OLED is made with aluminum as the cathode, poly[2-metoxy-5-(2-ethylhexyloxy)-1,4-phenylenevinylene] (MEH-PPV) as the active layer, poly(3,4- ethylenedioxythiophene)-poly(styrenesulfonate) (PEDOT:PSS) as an organic layer for hole transport and $Cu_{2-x}Se$ as an anode. Through the analysis of the obtained data and empirical investigations on the film conversion process, it was possible to discover an ideal time for the chemical bath of the electrode. Within 30 seconds of chemical bath to obtain the $Cu_{2-x}Se$ electrode, the OLEDs had charge injection only by the direct tunneling effect. Thus, it was shown that it is possible, through a chemical process, to change the $Cu_{2-x}Se$ electrode transport mechanism. To complete this work, EIS study was conducted to confirm the results obtained previusly.

Acknowledgments:

CNPq, CAPES, FAPEMIG, and FAPESP.

References:

[1] Silva et al., Advanced Electronic Materials vol.7, 2001189 (2021)

The influence of molecular geometry configuration shifting between RTP (Room Temperature Phosphorescence) to High Fluorescence Efficiency, in pure organic molecules

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This work aims to explore and to explain the photophysical behaviours in a series of pure organic molecules, which can exhibit room temperature phosphorescence (RTP) and high efficiency photoluminescence, by changing the moiety geometry position. These two interesting effects were observed in different molecules, room-temperature phosphorescence in air atmosphere and a high photoluminescence quantum yield (PLQY >70%), both properties are widely desirable, being RTP used as moieties for donor-acceptor molecules, bioimaging, and a high PLQY as a hyperfluorescence in active layer for devices. Experimental procedures were performed in order to fully determine the molecular photoluminescent properties, such as environmental effects, singlet-triplet alignment and S-T energy levels, charge transfer states, photoluminescence temperature dependence and photoluminescence lifetimes.

Acknowledgements:
CAPES, PGF-UNIFEI, FAPEMIG Project: APQ-00052-21 and APQ-04537-22 and UNIFEI

References:

[1] Ward, J. S.; Nobuyasu, R. S.; Batsanov, A. S.; Data, P.; Monkman, A. P.; Dias, F. B.; Bryce, M. R. Chem. Commun. 52, 2612-2615 (2016).

[2] Chan, CY., Tanaka, M., Lee, YT., Wong YW., Nakanotani H., Hatakeyama T., Adachi C., Nature Photonics 15, 203-207 (2021).

The Influence of Wetting on the Impedance Modulus of PVC/CNT Electrospun Membranes

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Membranes of PVC (polyvinylchloride) produced by electrospinning are useful for a variety of applications, such as air and water filtration systems, batteries, and protective clothing [1,2]. Among the characteristics of electrospun PVC membranes, it can be highlighted the mechanical resistance, the adjustable hydrophobicity, and the high porosity [1]. The combination of PVC with functional nanofillers, such as CNT (carbon nanotubes), might be used to further expand the use of the material [2]. The present study aims to evaluate the influence of wetting on the electrical impedance spectra for electrospun PVC/CNT membranes. PVC electrospun membranes containing up to 3 wt% CNT were prepared from THF/DMF solutions (18 wt% PVC, 20 kV, 15 cm NCD). They were characterized by FE-SEM and TEM in order to both analyze the fiber morphology and verify the dispersion of CNT inside the nanofibers. Finally, electrical impedance spectroscopy measurements were obtained for both dry and wet mats in the frequency range of 100 Hz to 1 MHz. No significant differences between impedance spectra and CNT concentration were found for dry membranes. On the other hand, a reduction in the impedance modulus from 8.1% (neat PVC) to 99.8% (3 wt% CNT) was observed for wet membranes. The last showed an exponential decrease of the impedance modulus with increasing the CNT concentration. This behavior might be related to the presence of carboxylic groups on the CNT surface, which enable proton conduction under wet conditions. TEM images show that most of the nanotubes are oriented along the fiber direction, while SEM micrographs evidenced membranes with homogeneous fiber distribution.

Acknowledgements:
Thanks to FAPESC for the financial support (2021TR928), UNIEDU for scholarship (18243), and the Multi-User Facility infrastructure from CCT/UDESC.

References:
[1] L.Q.Pham et al., Fibers 9, 12 (2021)
[2] L.Liu et al., Compos. Commun. 22, 100506 (2020)

THEORETICAL AND EXPERIMENTAL INVESTIGATION OF GAP ENERGY DEPENDENCE WITH THE INCREASE OF π-CONJUGATED SYSTEM IN BIS-CHALCONES

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Chalcones can present nonlinear activity: a substantial electron donor, a highly polarizable π-conjugated bridge, and a π-electron acceptor. Furthermore, a chalcone molecule with a π-conjugated system provides a significant charge-transfer axis with appropriate substituent groups on the terminal aromatic rings. Therefore, compared to conventional chalcones, bis-chalcones have a more extensive conjugate system, which results in a more significant overlap of molecular π orbitals, making them suitable for applications in optoelectronic devices. In this context, some parameters are essential, such as the electronic gap (E_g), the Highest Occupied Molecular Orbital (HOMO), and the Lowest Unoccupied Molecular Orbital (LUMO). Thus, the effects of increasing π-conjugation on decreasing theoretical and experimental gap energy for chalcones (1E,4E)-1,5-diphenylpenta-1,4-dien-3-one (DBA) and (1E,3E,6E,8E)-1,9-diphenylnona-1,3,6,8-tetraen-5-one (DBCN) synthed by the Claisen-Schmidt condensation method were investigated. Initially, conformers with minimum energy were geometry optimized through the Density Functional Theory (DFT) using the exchange-correlation functional M06-2X and 6-311+G(d,p) basis set. Then, we computed the vertical excitation energies and oscillator strengths to simulate the electronic absorption spectra (UV-Vis) keeping the same functional and basis set of the geometry optimization step. Finally, the Tauc graph was generated to compare the measurement of solid samples in diffuse reflectance performed in Shimadzu UV-2600 spectrophotometer using an integrating sphere ISR-2600Plus with $BaSO_4$ calibration standard.

Acknowledgment:
FUNCAP, CAPES, CNPq, and Profs. Valder Freire (UFC) and Hélcio Santos (UVA) for the support.

References:
[1]ZAINURI,D.A., et al. Acta Crystallographica Section E: Crystallographic Communications, v.74, p.1302-1308, 2018.
[2]DASILVA,P.S.P., et al. ChemPhysChem, v.19, p.82-92, 2018.
[3]IRFAN,A., et al. Optik, v.149, p.321-331, 2017.

Theoretical and Experimental studies of the Photophysical properties of novel and highly fluorescent difluoroboron flavanone β-diketonatecomplexes

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Difluoroboron β-diketonates complexes are highly luminescent with extensive properties such as their fluorescence both in solution and in solid state and their high molar extinction coefficients. Due to their rich optical properties, these compounds have been studied for their applications in organic electronics such as in self-assembly and applications in biosensors, bio-imaging and optoelectronic devices. The present work aims to study the photophysical and electrochemical properties of novel fluorescent difluoroboron flavanone β-diketonate complexes in different solvents and solid thin films [1]. The photophysical studies revealed that the substituent groups and the solvent media significantly influencethe electrochemical and photophysical properties of the final compounds, especially the molar absorption coefficient, fluorescence quantum yields, and the band gap. Moreover, thecompounds exhibited a single excited-state lifetime in all studied solvents. In solid films, theabsorption bands of the compounds showed a red shift when compared to absorption spectra in solution and the excited state decays of the films showed reduced lifetime, suggesting J-type aggregation of molecules. Electronic structure calculations using DensityFunctional Theory (DFT) and Time-dependent density functional theory (TDDFT) were performed to assess ground and excited state properties of the compounds and determinetheir theoretical absorption spectra.

Acknowledgements:

CNPq, FAPEMIG

Reference

[1] Elida Betania Ariza Paez, Sergio Curcio, Natália P. Neme, Matheus J. S. Matos, b Rodrigo S. Correa,a Fabio Junio Pereira, Flaviane Francisco Hilário, Thiago Cazati and Jason Guy Taylor. New J. Chem., , 44, 14615--14631 (2020)

Two-photon absorption and dynamics of excited statesin Bromo-Chalcone derivatives

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Chalcones are a family of organic compounds that exhibit good nonlinear optical properties [1]. Additionally, they have a relatively simple synthesis and various pharmacological properties, making them promising subjects of study, with potential applications in areas such as bio-photonics. In this work we study the dynamics of excited states and the effect of substituents (electron donors and acceptors) on the two-photon absorption (2PA) cross-section of bromo-chalcone derivatives using the z-scan technique [2]. Nonlinear optical experiments are performed using 200-fs pulses from a YGW laser system, which, when coupled with an optical parametric amplifier, allows for tuning the excitation wavelength from the UV to the near-infrared. Therefore, the 2PA cross-section spectrum is obtained, enabling to correlate the nonlinear optical response with the molecular structure. Initially we have selected a few chalcone compounds, which were diluted in DMSO, and their linear absorption and fluorescence spectra have been measured. It was observed that the samples exhibited two UV absorption bands, except for the one with the amino group (-NH2), which showed a significant shift of the first band (now centered at 432 nm). Such a molecule was also the only one that exhibited fluorescence, centered at 558 nm. Yet, from the open aperture (absorptive) z-scan measurements with this sample, it was observed that the transitions allowed by one photon were also allowed by two photons, which is a characteristic of a non-centrosymmetric molecule. Further studies are being performed with the other chalcone compounds to correlate the nonlinear spectrum and specific molecular features.

References:
[1] Muhammad Nadeem Arshad et al. Synthesis, crystal structures, spectroscopic and nonlinear optical properties of chalcone derivatives: A combined experimental and theoretical study. [2] M. Sheik-Bahae et al "Sensitive measurement of optical nonlinearities using a single beam"

Two-photon absorption process in symmetric diketopyrrolopyrrole derivatives: Influence of Thienyl Moiety and Bromine Atom

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In recent years, the search for novel organic materials capable of absorbing two photons has grown due to their potential applications in three-dimensional optical data storage, microfabrication, high-resolution three-dimensional imaging of biological systems, optical power limitation, and photodynamic therapy [1]. In addition, the two-photon absorption (2PA) process is an important tool for understanding the relationship between electronic/molecular structure and optical properties at the molecular level [2]. Among various organic compounds, diketopyrrolopyrrole derivatives, also known as DPPs, have shown remarkable properties, such as an accessible synthesis route, easy modification by incorporating substituents, high fluorescence quantum yield, and planarity. Such features makes DPPs an extremely interesting structure. However, there is little information about the nonlinear optical properties of DPPs. Here, a complete characterization of two symmetric dithienyl-diketopyrrolopyrrole derivatives was carried out, exploring the influence of the thienyl moiety (an electron-rich structure acting as an extension of the π-conjugation) and the bromine atom in the 2PA process. The results were quite promising, with both compounds exhibiting a 2PA band in a dark 1PA spectral region, as expected due to the dipole selection rule. The substitution of hydrogen atoms with bromine led to 1.4 times increase in the 2PA cross-section value. This effect was associated with a greater electronic delocalization observed in the structure containing the bromine atom. This showsa possible path towards the architecture of molecules with larger nonlinear linear responses.

[1] Valverde et al., J. Mol. Liq., v. 373, p. 121250, 2023;
[2] Xu et al., J. Mater. Chem. C., v. 9, p. 1520-1536, 2021.

Unraveling the ICT character in D-A copolymers: an experimental and theoretical approach.

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The donor–acceptor (D–A) approach in the conjugated polymer is an effective method to control the energy levels of the material. For D–A copolymers, the first photoexcitation is expected to correspond to a state where the excited electron is promoted to the first unoccupied orbital, which is mainly localized at the acceptor moiety, while the hole is localized at the donor unit. This characteristic is usually named as internal charge transfer (ICT), an intrachain electron transfer from the electron-rich to the electron-deficient unit. In this work, D–A copolymers composed of diketopyrrolopyrrole (DPP), thiophene (Th), benzothiadiazole (BT), and fluorene (F) as a building block have been synthesized and analyzed in terms of optical properties and theoretical studies. For these, two D-A copolymers, PFBT and PFThDPPTh, were synthesized using Suzuki coupling. The electronic spectra of both copolymers exhibited the dual-band absorption profile (λmax in 317 and 445nm for the PFBT and 370 and 650 nm for PFThDPPTh), suggesting that the ICT processoccurs in both structures. However, our results using density functional theory (DFT) andtime-dependent DFT (TD-DFT) frameworks contrast with what is often observed and reported for other D–A copolymers. For the PFThDPPTh, the theoretical results pointed outthat the HOMO has a major contribution from DDP, while in the LUMO, DPP and Th havesimilar contributions but are still being dominated by DPP. This is in stark contrast withwhat has been reported regarding D–A copolymers. We note that, until now, the designation of a given copolymer as D–A is quite cloudy, based primarily on thevisualization of electronic absorption spectra. In this concern, our results demonstrate that the analysis of the composition of the near gap states is crucial to attribute the character ofa given copolymer, avoiding misinterpretation of the results.

Acknowledgements:
INEO, CNPq and Fundação Araucária.

Use of spectroscopy and chemometrics in the study of pen inks applied to Forensic Documentoscopy

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Use of spectroscopy and chemometrics in the study of pen inks applied to Forensic Documentoscopy
Forensic Sciences have been following the evolution of technical-scientific knowledge and there are currently several areas of specialization in Criminalistics. Among them, we can mention Forensic Documentoscopy, which consists of a set of specialized knowledge aimed at studying and interpreting the material traces of document forgeries and alterations, which may be of interest to the clarification and proof of factual issues, at the service of justice [1]. In this sense, the traces associated with pen inks present in documents collected at a crime scene are object of study of Forensic Documentoscopy, since it allows determining the veracity or otherwise of documents for judicial purposes. Given the need to standardize the operational procedures for document analysis at the Forensic Institutes, Forensic Scientist must develop a protocol for pen ink analysis. In this work, we aimed to combine spectroscopic methods, such as Raman and FTIR, with chemometric methods such as PCA and PLS to allow discriminating manual releases of 62 pen inks of different brands in blue and black colors sold in the national territory in a fast, safe and efficient way. Preliminary results indicated that the combination of spectroscopies UV-VIs and FTIR with PCA enabled the distinction between the types of pen inks available in Brazil in a significant way.

Acknowledgments:To the Physics Institute of UnB, the Forensic Institute of PCDF, the Research Support Foundation of the Brazilian Federal District (FAP/DF) and the National Council for Scientific and Technological Development (CNPq).

References:

[1] Eraldo Rabello.Curso de criminalística: uma sugestão de programa para as Faculdadesde Direito. Porto Alegre, Sagra－Dc Luzzatto, ISBN: 8524104910, 1996.

Volumetric capacitance in organic electrochemical transistors: Fundamentals and guidelines on how to correctly measure it experimentally

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Organic electrochemical transistors (OECTs) were first introduced in 1984. However, only in the late 2000s did these devices gain notoriety. In 2015, a big step was taken in understanding OECTs when it was found that capacitance scales with channel volume [1]. In 2016, the so-called volumetric capacitance (C*) was microscopically explained as the product of many parallel plate capacitors between the polymer chain and the solvated ion[2]. Finally, in 2017, C* was established as one of OECT's benchmark. Despite that, the extraction of C* can still be controversial. Usually, it is done by means of electrochemical impedance spectroscopy (EIS). Being a two-terminal technique, the drain and source contacts must be shorted and grounded together. This mimics the OECT operation only when V_D is zero, neglecting any different operation mode or the non-uniform doping profilethroughout the channel. Even so, EIS has been extensively used by the OECT community. The EIS limitation can be overcome by measuring the drain-transient current under a pulsedV_G and analyzing the data in the light of the Faria-Duong model [3]. Being three-terminal based measurements, the drain-transient technique allows one to apply V_D while pulsing V_G. Here, we conducted a thorough comparison between the two techniques, using PEDOT:PSSbased OECT and showing that EIS can be considered for extracting C* only when $V_G >> V_D$. For any other case, only the drain-transient technique provides manners to evaluate the dependency of C* with V_D. By comparing results obtained from both techniques, guidelines for a definitive way of measuring C* in OECTs are discussed and established.

Acknowledgements

We would like to acknowledge CAPES, FAPESP and INEO.

References

[1] J. Rivnay, et al. Sci. Adv. 2015, 1, e1400251

[2] C. M. Proctor, et al. J. Polym. Sci. Part B: Polym. Phys. 2016, 54,1433

[3] G. C. Faria, et al. Org. Electron. 2017,45, 215

S-Eco-design Strategies of Bio-Polymeric/Nanocellulose Materials: from fundamental aspects to marketing

Bacterial cellulose, fibroin and other biocompatible substrates for organic devices.

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Recently, great effort has been made to develop both flexible and conformable electronic devices, either as a way to improve ergonomics, making them fit the human body or even to prevent them from breaking under pressure. The search for cheap, sustainable and, mainly, biocompatible materials becomes necessary, since such devices would be subject to long exposure to living tissues. So far, together with the Organic Light Emitting Diodes (OLEDs) and Organic Solar Cells (OSC), there is a demand for new and efficient organic field-effect transistors (OFETs) on biocompatible substrates to allow conformable organic electronics. For the OLEDs great efforts have been reported in the literature to use them in the biomedical field, such as in Photodynamic Therapy. Concurrently, there is a huge interest in the design of advanced materials devised from natural sources. Bacterial nanocellulose (BNC) is a natural polymer synthed by many microorganisms, notably by non-pathogenic strains of ***Komagataeibacter***(***K. xylinus, K. hansenii, K. rhaeticus***). BNC shows distinct physical and mechanical properties make BNC ideal for many areas, including sensors, drug delivery, wound healing, and as conformable/flexible substrates for organic electronics. Other examples of the materials that can be used for this purpose are fibroin, which is a natural biocompatible material extracted from the silkworm cocoon and a new biopolymer obtained from onion pulp (***Allium cepa L.***). BC can be processed with fibroin in order to increase the substrate' s transparency, while cellulose makes fibroin more malleable and robust. Moreover, indium tin oxide (ITO) and SiO_2thin films need to be deposited onto these bio-substrates to obtain conformable, transparent and conductive anodes on top of which organic devices were produced. Here, we review the production methods of these substrates, their properties and applications, focusing on electronic organic devices, especially OLEDs and OFET.

Bacterial celulose. From biomedical to photonics applications

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Bacterial cellulose or biocellulose (BC) comprises homopolymer of D-glucopyranose residues bonded through β-(1 → 4) glycosidic linkages usually produced by Gluconacetobacter genus. The BC hydrated membranes consist of a three-dimensional network of entangled cellulose nanofibrils, which provides highly porous structures, high surface area, and high purity (lignin- and hemicellulose-free). This cellulose source displays interesting optical, mechanical and chemical properties making it a wonderful material with applications ranging from biomedical (bandages for wounds, burns, templates for bones regenerations and ophtalmological lenses) to photonics (substrates for flexible OLEDs and flexible Random Lasers, photochromic materials, liquid crystalline selfsustainable films, aerogels photocatalysts) [1-3]

All these applications have been touched by our group in the last years and the main results, conclusing remarks and perspectives will be presented.

Acknowledgements: Brazilian agencies, CNPq, CAPES, FAPESP and the National Institute of Photonics are acknowledged for the support.

References:
[1]-T.A. Faraco et al, 1,2, Review of Bacterial Nanocellulose as Suitable Substrate for Conformable and Flexible Organic Light-Emitting Diodes, Polymers 2023, 15, 479
[2]-M.V. Santos, Cellulose Based Photonic Materials Displaying Direction Modulated Photoluminescence, Frontiers in Bioengineering and Biotechnology, 9, 617328, 2021
[3]-E.P. Ferreira-Neto et al, Bacterial Nanocellulose/MoS2 Hybrid Aerogels as Bifunctional Adsorbent/Photocatalyst Membranes for in-Flow Water Decontamination ACS Appl. Mater. Interfaces 12, 41627, 2020

Bacterial nanocellulose: From fundamental aspects to marketing

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Bacterial nanocellulose (BC) has received considerable attention in the last years. The unique properties of BC, such as high purity, mechanical stability, thermostability, crystallinity, and biocompatibility make it a promising candidate for commercial applications in different areas (food, electronics, bioengineering, cosmetics and biomedical areas). BNC can be associated with organic/inorganic entities to produce new organic-inorganic nanocomposites (OIN). Using different pathways named bottom-up approach and top-down approach, biopolymer-based OIN can be applied to tailored functionalities and emerging applications. In this lecture I will explore all-BNC products (hydrogels, drug delivery films, food applications, e.g), and I will point out the possibility to increase strength cooperation between academia and industry in the formation of new Start-ups and joint ventures.

Acknowledgments
The author thank CAPES, CNPq, Embrapi, Finep, INCT/Polissacarídeos, LNLS and Fapespn° 2018/25512-8 for the financial support.

Bio-polymer based hybrid materials as functional adsorbents and delivery tools.

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Hybrid organic-inorganic materials have broad perspective of application due to combination of functional components. Cellulose is the most abundant bio-polymer that can easily be transferred into nano form. Its surface modification facilitates creation of hybrid materials that can incorporate both organic and inorganic functional components. Cotton-based nano cellulose terminated by carboxylic acid functions possesses strong affinity to metal oxide particles. As nano particles for grafting one can choose either metal oxides strongly binding bio-active molecules/pharmaceuticals for drug delivery, such as TiO_2 [1,2], or oxides possessing important physical (optical or magnetic) characteristics, such as FeO_x [3]. Produced composites display ability to quickly and efficiently uptake several classes of important pharmaceuticals and to deliver them in a chemically or physically controlled way [3,4]. Chemical surface modification with grafting specific ligand functions opens for creation of selective adsorbent materials for recycling of critical metals. Their adsorption and desorption mechanisms have been investigated gaining insights from structural characterization of molecular model compounds [5]. Proposed approaches open prospects for development of circular economy and finding environmental solutions for sustainable future.

Acknowledgments

The authors would like to thank the ÅForsk foundation, grant No. 21-75 and Swedish Council for Internationalization of Higher Education and Research (STINT) grant BR2019-8506 for Brazilian-Swedish collaboration.

References

[1] Galkina, O.L., Seisenbaeva, G.A., et al. J Mater. Chem. B. 3, 7125-7134 (2015).
[2] Evdokimova, O.L., Seisenbaeva, G.A., et al. Nanomaterials 8, 00228 (2018).
[3] Breijaert, T.C., Seisenbaeva, G.A., et al. Carbohydrate Polymers 291, 119560 (2022).
[4] Gaio, S., Seisenbaeva, G.A., et al. Mater. Adv. 3, 7228-7234 (2022).
[5] Breijaert, T.C., Seisenbaeva, G.A., et al. Dalton Trans. 51, 17978-17986 (2022).

Cellulose nanocrystals and composites: from synthetic approaches to engineering materials

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Cellulose nanocrystals (CNC) in the form of a stable aqueous suspension, self-assemble intoa liquid crystalline (LC)[1] phase giving rise to solid structures with tunable structural coloration[2]. The chemical versatility of cellulose also leads to a well-known variety of derivatives that can present LC phases. This feature that also impart distinct and interesting properties to the final product but is less exploited in creating new materials. In this small overview, the bio-based polymers on the development of innovative materials are shown. Such as, all-cellulosic composites of CNCs/hydroxypropylcellulose that can create photonic structures that reflect light from UV to NIR wavelength[3]. If microgaps, found within the cross-section of CNCs films, are infiltrated with a nematic liquid crystal, a tunable left- and right-handed CPL system is achieved[4]. Hydroxypropylmethylcellulose in a LC phase displays traveling colored patterns that change the sign of the helix pitch[5] with time. CNCs can also act as matrices for magnetic iron nanoparticles or reinforce chitosan hydrogels for biomedical applications. These examples open new horizons for the application of these renewable materials.

Acknowledgments

The authors acknowledge the support of FCT-Fundação para a Ciência e a Tecnologia, I.P.,in the scope of the projects LA/P/0037/2020, UIDP/50025/2020 and UIDB/50025/2020 of the Associate Laboratory Institute of Nanostructures, Nanomodelling and Nanofabrication-i3N, and also support from Action European Topology Interdisciplinary Action (PhoBioS CA21159).

References

[1] da Rosa, R.R.; Adv Mat, 34, 2108227 (2022).
[2] Fernandes et al. Curr Opin Solid State Mater Sci, 23, 63 (2019).
[3] Saraiva et al. Crystals, 10, 122 (2020).
[4] Fernandes et al. Adv Mat, 29, 1603560 (2017).
[5] Silva et al. Commun. Mater. 2, 79 (2021).

Cellulose nanofibers-based composites for applicationas electronic devices

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The use of plant sourced cellulose nanofibers (CNF) to fabricate sustainable and high performance materials is foreseen as a green solution in some areas, for example in the electronic industry and water treatment [1-3]. CNF give adequate H-bonding between the nanofibers resulting in superior mechanical properties, so that composites may be easily produced in the form of resistant and flexible films from the mixture of CNF as a matrix and other components dispersed into it. In this way, we have prepared different composites: i) mixing CNF with conducting polymers polyaniline (PANI) or polypyrrole (PPY), graphene oxide (GO) and magnetic manganese ferrite nanoparticles (MNP) to produce a conducting composite that was tested as electromagnetic shielding; ii) CNF with carbon nanotubes (CNT) in order to investigate their electrochemical properties in view their use in energy storage devices. The effectiveness of electromagnetic shielding in the range of 8.2-12.4 GHz for each of the conductive composites were evaluated and the maximum shielding efficiency at 8.2 GHz in both polypyrrole (-9.14 decibels) and polyaniline (-6.81 decibels) films occurred in films with the highest conductivity values. For the CNF and CNT composites, the cyclic voltammograms obtained for the composites with different percentages of CNT showed a capacitive profile, with the profile obtained for the composite of 50% (m/m) of CNT in relation to CNF, characteristic of supercapacitors with pseudocapacitance. These studies corroborate the versatility of using CNF as a support for new materials with innovative applications.

Acknowledgments

The author express especial acknowledgements to the Foundation of Support Research of the State of Minas Gerais (FAPEMIG), the National Council for Scientific and Technological Development (CNPq), and the Coordination for the Improvement of Higher Education Personnel (CAPES) for financial support.

Cellulose nanomaterials and advanced applications

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There has been an explosion of interest in the use of biomass as a source of renewable energy and materials. This has led to a concurrent increase in the research activity on the biosynthesis, isolation, and properties of cellulose, the key structural component in plant cell walls. One focus of this activity has followed from the recognition that, by suitable chemical and mechanical treatments, it is possible to produce fibrous materials with one ortwo dimensions in the nanometer range from many naturally occurring sources of cellulose. As is usually the case when a new field is developing, the terminology is somewhat confused, but the term "nanocellulose" is now used to cover the range of materials derivedfrom cellulose with at least one dimension in the nanometer range. Nanocellulose-based materials are carbon neutral, sustainable, recyclable, and nontoxic; they thus have the potential to be truly green nanomaterials with many useful and unexpected properties. What is not to love? Multiple mechanical shearing actions can be used to release more or less individually cellulose microfibrils. This material is usually called microfibrillated cellulose. Longitudinal cutting of these microfibrils can be achieved by submitting the biomass to astrong acid hydrolysis treatment, allowing dissolution of amorphous domains. The ensuing nanoparticles occur as rod-like nanocrystals. Similar acidic treatment carried out on starchgranules allows obtaining platelet-like nanoparticles. Impressive mechanical properties andreinforcing capability, abundance, low weight, and biodegradability of these nanoparticlesmake them ideal candidates for the processing of polymer nanocomposites. With a Young' smodulus around 130 GPa and a surface area of several hundred m2.g-1, they have the potential to significantly reinforce polymers at low filler loadings. However, as for any nanoparticle, the main challenge is related to their homogeneous dispersion within a polymeric matrix.

Ecodesign-Inspired Nanocellulose Production: High Efficiency and Customizable Properties with Enzyme-Based Platform Technologies

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Enzyme-based technologies can play a significant role in ecodesign for the production of cellulose nanoparticles. This is because ecodesign aims to optimize industrial processes to minimize their environmental impact, while enzyme technologies offer several advantages over traditional chemical and mechanical processes in terms of their environmental friendliness and efficiency. Our efforts have been focused on developing enzyme-based platform technologies that are not only viable, robust, and scalable but also eco-friendly for manufacturing nanocelluloses of different grades suitable for various applications. In this presentation, we will show results to demonstrate that ecodesign-inspired nanocellulose production from hardwood Kraft pulp with enzyme-based platform technologies is an efficient strategy to produce cellulose nanocrystals (CNCs), cellulose nanospheres (CNSs), and cellulose nanofibrils (CNFs) with high yield. It will also cover aspects of how the enzymatic approach can also be designed to tailor the properties (i.e., morphology, surface chemistry, rheological behavior, chemical composition) of cellulose nanoparticles to specific applications.

Acknowledgments: São Paulo Research Foundation (FAPE grant number 2021/07023-2).

Electric forces as a tool for manufacturing nanostructured nanocellulose materials: thermoplastic nanocomposites, thermoformable panels and continuous filaments

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Electrical charges can be useful to produce stable colloidal suspensions due to dominant repulsion forces between particles, as well to produce the opposite effect leading to coagulation and then flocculation of suspensions or to induce order as a driving force to self-assembly. Nanocellulose suspensions can exist as non-charged or charged nanofiber suspensions. Here we discuss the use of such charges to produce very homogeneous nanocellulose composites in non-polar vinyl polymers [1], thermoformable panels of nanocellulose and vinyl polymers [2] and continuous fibers from interfacial complexation of anionic surface charged nanocellulose and cationic natural polyelectrolytes [3]. Thermoplastic nanocomposites and thermoformable panels are produced by the combination of polymer nanoemulsions and nanocellulose suspensions. The final product is produced during coalgulation and flocculation induced by charge neutralization of the polymer emulsion with inorganic coagulants and by the bridging effect induced by nanocellulose which works similarly to non-charged or very low charge high molecular weight polyelectrolyte. The filaments are produced by complexation of a cationic high molecular weight polyelectrolyte such as chitosan and negative charged nanocellulose that again works in a similar manner as a high molecular weight anionic polyelectrolyte.The authors acknowledge to CAPES and CNPq.References:[1] Carvalho, A.J.F.; Trovatti, E.; Casale, C.A., J. Mol. Liq., 272, 387-394 (2018).[2] Chiromito, E.M.S.; Trovatti, E.; Carvalho, A.J.F., J. Renew. Mater., 7(5), 403-413, (2019)[3] Grande, R., Trovatti, E., Carvalho, AJF and Gandini, A., J. of Mater. Chem. A, 5, 13089 - 13103 (2017).

Nanocellulose materials for 2D and 3D applications

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Processing nanocellulose structures with complex architectures and high cellulose contents is chal-lenging. This can sometimes prevent nanocellulose-based synthetic materials from reaching the level of structural complexity and mechanical properties observed in their biological counterparts, such as wood and plant tissues. At Empa, we demonstrate various approaches to manufacture nanocellulose composites, hydrogels and aerogels with high degree of particle alignment and shape fidelity. We prepare soft and stiff composites by our 3D printing methods. Such composites exhibit micro-structures with a high degree of particle alignment and spatially tailored mechanical properties. These materials may find applications in cellulose-based structural, biomedical, soft robots and energy-related products.

Nanostructured Polysaccharides Materials for Water Purification

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The design and synthesis of nano-architected cellulose can act as efficient materials for water purification. The talk aims to study and compare the performance of a polyvinylidene fluoride (PVDF) electrospun membrane, unmodified cellulose nanofiber (CNF) based PVDF membrane, and Meldrum's acid (2,2-dimethyl-1,3-dioxane-4,6-dione) modified CNF-based PVDF membranes against the Fe2O3 nanoparticle filtration and crystal violet (CV) dye adsorption. Herein, we introduced a facile method to produce a unique green adsorbent material from cellulose nanofibers (CNFs) via a nonsolvent assisted procedure using Meldrum's acid as an esterification agent to enhance the adsorption toward positively charged crystal violet dyes. Most of the surface modifications of CNFs have been done using toxic organic solvents like pyridine, dimethyl acetate, (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO), etc. So far, this is the first report on the surface modification of cellulose nanofibers via a nonsolvent assisted procedure. Both CNF-based PVDF membranes were prepared by successive coating of modified and unmodified CNFs on to the surface of a PVDF electrospun membrane. All the demonstrated membranes showed high filtration capacity against the Fe2O3 nanoparticles. With the 10 mg/L of crystal violet (CV) aqueous solution, CV adsorption of PVDF electrospun membrane, and unmodified CNF-based PVDF membrane was around 1.368 and 2.948 mg/g of the membrane respectively, whereas it was 3.984 mg/g of the membrane by Meldrum's acid CNF-based PVDF membrane. The demonstrated Meldrum's acid modified CNF-based PVDF membrane was proven to be the efficient media that can concurrently eliminate the Fe2O3 nanoparticles and CV dyes from the water. The investigation into the surface chemistries of CNFs beyond the adoption of toxic solvents can enhance the economic usefulness of the process and also yield a new eco-friendly adsorbent material that is agreeable to adsorbing various toxic pollutants.

Surface-engineered nanocellulose towards improvedkinetic stability of Pickering emulsions

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Surfactantless yet metastable multiphase systems are increasingly arousing scientific and technological interest. In particular, when stabilized by solid particles sitting at the oil/water interface, Pickering emulsions offer high kinetic stability and tunable rheological behavior. We herein isolated cellulosic nanostructures from biomass engineered their surface chemistry towards increased interfacial activity. Cellulose nanofibrils (CNF) were regioselectively esterified via imidazole-catalyzed reactions using acetic or isobutyric anhydrides. Introducing the hydrophobic ester moieties −acetyl and isobutyryl, respectively− at functionalization degrees of 0.6 to 1.0 mmol g-1 −confirmed by NMR, ATR-FTIR, and inverse gas chromatography− altered the rheological profile of sunflower oil-in-water Pickering emulsions and led to superior metastability. The interfacial energy betweena drop of aqueous CNF suspension and sunflower oil was reduced from 24 ± 1 mN m−1 byca. 15% (unmodified CNF), 22% (acetylated CNF), and 20% (isobutyrylated CNF). Likewise,the contact angle between water and films cast from the aforementioned CNF samples was increased from 26º to 52º and 61º, respectively. While our surface hydrophobization approach might stand by itself for O/W emulsions, our group took advantage of the netnegative charge of the oil droplets to promote its electrostatic complexation with cationicCNF decorated with quaternary ammonium moieties. We show that more than a single mechanism is at play when kinetically stabilizing Pickering emulsions using nanocellulose, at least chemical compatibility, electric charge, and continuous phase thickening.

Acknowledgements:
FAPESP (grant no. 2021/12071-6), CNPq (grant no. 304753/2022-0), and CAPES (Finance Code 001).

Use of algae biomass to obtain nanocellulose and technological applications

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Currently, the use of renewable resources such as biomass has been increasing [1]. Seaweed is one of those renewable resources that have polysaccharides, lipids, vitamins and minerals in their cellular structures. However, only 50% of the various existing species have been identified and studied [2]. This characterizes algae biomass as an important raw material for several technological applications. Among the materials that make up this raw material, cellulose is undoubtedly of great interest, as it is widely used for various industrial applications. Since then, it has received great attention mainly due to its economic advantage. The extraction of cellulose fibers from algae has been considered a great alternative in the context of environmental maintenance, and has support for the growing market demand [3]. In the last two decades, nanocellulose has received much attention due to its renewable nature and properties such as biodegradability, biocompatibility, mechanical strength, lightness and high surface area [1]. Therefore, in this study, it is proposed the extraction of nanocellulose from macroalgae and characterization regarding its chemical and morphological structure. The results of Fourier transform infrared spectroscopy revealed that the employed methodology was effective for obtaining nanocellulose. The electron microscopy images showed the presence of fibers with a diameter on the nanometric scale.

References

[1] Paniz, O.G., Pereira, C.M.P., Pacheco, B.S. et al. Cellulose 27, 113-126 (2020)
[2] Sanzo GD, Mehariya S, Martino M, et al. Mar Drugs. 16(9):334 (2018)
[3] Filho, A.V, Santana, L.R. et al. Algal Research 71, 103084 (2023)

Cellulose/Graphene Oxide Composites: rheological characterization of dispersions and mechanical properties of selfsustained films

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There is much interest in developing environment friendly packaging materials, especially considering the spike in e-commerce, delivery services, and the use of single-use disposable packages in the last years. Cellulosic materials allow for the production of strong biodegradable films, while further contributing to the circular economy when sourced from regional crop byproducts.[1] In this work, composite dispersions of nanofibrillated cellulose and graphene-oxide were prepared by microfluidization, after the TEMPO-mediated-oxidation reaction of cellulose pulp extracted from sugarcane bagasse. Rheological tests in oscillatory and permanent regimes indicated that the composite dispersions presented thixotropic gel-like behavior. It was possible to produce uniform selfsustained composite films from tape-casting process, with graphene-oxide content of 1 and 5 wt%. Tensile testing showed that the presence of graphene-oxide enhanced the mechanical properties of the films. The addition of 1 wt% of graphene-oxide content doubled the elastic modulus value and increased the tensile strength of the films in 85%. The mechanical enhancement is attributed to the formation of an interconnected structure between the nanocellulose fibers and the graphene-oxide.[2]

Acknowledgements:
National Council for Scientific and Technological Development (CNPq) grant no. 140459/2018-9.
Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES)References:

[1] A. Dufresne, Mater. Today., 16, 220-227 (2013)
[2] M. Achaby, N. Miri, A. Snik, M. Zahouily, K. Abdelouahdi, A. Fihri, A. Barakat, A. Solhy, J. Appl. Polym. Sci., 133, 42356 (2016).

Exploring the Influence of Freezing and Drying Processes on Nanocellulose-Natural Rubber LatexFillers for Enhancing Mechanical and Physical Properties of Bionanocomposites

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The excessive use of petroleum-based plastic packaging has raised concerns about its environmental impacts, leading to research on sustainable and economically viable bionanocomposites. However, there are still challenges related to the intrinsic characteristics of some biopolymers that differ substantially from polyolefins, in terms of mechanical properties and water affinity. To address these issues, cellulose nanocrystals (CNCs) functionalized with natural rubber latex (NR) were explored as an effective alternative to enhance mechanical and physical properties of biopolymers. The functionalization process was developed by applying the freeze-drying method, which was inspired by the ice-templating approach to obtain nanocellulose-based aerogels. The effectof the functionalized nanofiller mass percentage in the biopolymer matrix, the NR content inthe nanofiller, and the nanofiller preparation method were evaluated for their impact on the mechanical and water vapor permeability (WVP) of bionanocomposites. The bionanocomposite films exhibited visible light transparency and mechanical properties comparable to conventional packaging such as polyethylene and polypropylene. The films also exhibited low WVP, indicating that they could serve as a significant barrier to other gases. These findings indicate a promising avenue for the development of a new generation of green nanofillers that can be conveniently hydrophobically tailored to suit various biopolymer matrices via different processing methods. Acknowledgement Fapesp process 20/08651-4.

Large-scale graphene synthesis using lignin asprecursor

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In this work, a new approach to the synthesis of lignin-derived nanocarbon crystals based on lignin depolymerization is proposed. The lignin chemical structure was modified through a controlled routine, which was based on sequential UV light irradiation, hydrothermal carbonization in autoclave, vacuum degassing, pyrolysis and mechanical exfoliation [1]. After these steps, it was obtained a lignin-based nanocarbon with a sp2-hybridized nanoporous framework structure. The used catalyst-free route resulted in high nanocarbon yields and turns out to be an eco-friendly strategy of a synthesis of low-cost carbon material. The characterization of the lignin-derived nanocarbon was carried out using X-ray diffraction measurements, infrared, Raman and X-ray photoelectrons spectroscopies analyses, while its morphology was evaluated by scanning and transmission electron microscopy. Chemical spectroscopic and microscopy studies showed that the pre-treatment steps were crucial to produce, after the pyrolysis, a lignin nanocarbon phase composed of ordered structural domains with the apparent lamellar morphology of randomly oriented carbon sheets. In contrast, the final material of not pretreated samples, after pyrolysis, consisted of a mixture of ashes and lignin nanocarbon with much more disordered structure. We believe that the approach to lignin’s treatment proposed here has good prospects as a large-scale production method of a material similar to reduced graphene oxide. The proposed approach may be adapted to various biomass feedstocks with aromatic groups’ content suitable for their high yield polycondensation.

Acknowledgments: Faperj (grants E-26/201.424/2022, E-26/211.113/2019, E-26/202.746/2018), CNPQ (grant 427792/ 2018-5) and Finep (grant 29375).

References:

[1] D. Rocha a, J. Luzardo, D. Aguiar, S. Massafra, R. Valaski, A. Silva, E. Ferreira, R. Simão, B. Archanjo, A. Kuznetsov, J. Araujo, Carbon, 203, 120-129 (2023).

Modified matrices of gellan gum doped with lanthanide ions for the production of sensors by 3D printing

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Gellan Gum (GG) is a natural polysaccharide that can be modified to suit different applications [1], including the production of biocompatible hydrogels. In this work, GG was combined with sericin (SS), a protein that is a residue of fibroin extraction in silkworm cocoons, to create a functional composite. This material showed interesting mechanical and optical properties, such as the ability to reproduce patterns on its surface and angle-dependent iridescent colors, which make it useful for various applications such as sensors, random lasers, and structural color systems [2]. In parallel, the GG matrix is being modified with 3-glycidyloxypropyltrimethoxysilane (GPTMS) in order to enable the gelation of this hybrid system via photopolymerization. Both modified systems were doped with lanthanide ions, important in the development of photonic devices due to their distinguished luminescent properties. These interactions with Eu^{3+}ion showed an interesting sensitization effect, resulted in an increased emission intensity and longer lifetime of the excited state [3]. Additionally, 3D printing technique is being used to manufacture three-dimensional multilayer structures from these photopolymerizable materials, allowing for the creation of high-precision sensors and other devices with a wide range of applications in nanotechnology.

Acknowledgements: FAPESP (2019/18828-1), CNPq (154100/2022-6) and the Universidade de São Paulo (USP) for financial support.

References: [1] Bacelar, A. H. et al. J Mater Chem B, v. 4, n. 37, p. 6164-6174 (2016). [2] Perry, H. et al. Adv Mater, v. 20, p. 3070-3072 (2008). [3] Binnemans, K. Coord. Chem. Rev., v. 295, p. 1-45 (2015).

Non-toxic Aerogel Comprising Nanocellulose and Natural Rubber Latex for Removal of Heavy Metal in Aqueous Environmental

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Nanocellulose-based aerogels have been reported as an eco-friendly alternative for water remediation (organic pollutants, heavy metal ions, and dye, e.g.) due to their highly porous network, large surface area, renewability, and biodegradability. However, cellulose nanofibrils (CNF) structures are unstable in aqueous media due to their high hydrophilicity, demanding chemical (or physical) crosslinking to maintain aerogel structure to avoid disassembling of aerogel 3D network in water. To overcome these drawbacks, natural rubber latex (NR) has been incorporated into CNF porous structures to improve wet resilience, enhancing structural resistance in aqueous media. Thus, aerogels comprising CNF and NR were developed. The heavy metal adsorption and CNF/NR ecotoxicity were assessed herein. Oxidated CNF-isolated from sugarcane bagasse was mechanically blended to NR to produce oxi-CNF/NR-based aerogels physically crosslinked with NR particles. The oxi-CNF/NR aerogels were obtained through the ice-templating protocol. They exhibited heavy metal adsorption capacity ranging from 108 to 420 $mg \cdot g^{-1}$, showing ~70 % removal capacity over the reuse cycles.[1]Furthermore, the ecotoxicity tests indicated, for the first time, not only that oxi-CNF/NR effectively remediated *Daphnia smilis*reconstituted water contaminated with Cu (II) and mitigated the toxic effect of Cu (II) at exposure over 60 ugL-1 close to the environmental scenario, but also the non-toxicity of oxi-CNF/NR by itself. Thus, these findings show oxi-CNF/NR aerogels as a promising, non-toxic, eco-friendly alternative for environmental remediation. Acknowledgement Fapesp process 2020/08651-4.

Spectroscopic studies of a composite from Eu^{3+}-doped cellulose nanocrystals/fibroin for luminescent cholesteric film production

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The synergy of the luminescent behavior of lanthanide ions (Ln^{3+}) with the physicochemical properties from cellulose nanocrystals (CNC), i.e., crystalline domains with strong intermolecular interactions [1], and silk fibroin, a protein with excellent mechanical and optical properties [2], play an important role on the development of new functional materials and photonic development. In this work, different routes to obtaining CNC were studied; in addition, the interaction of Eu^{3+}allowed us to study the coordination environment, aiming the production of different structural materials in order to generate photonic devices with color tunable in response to external action [3]. The objectives of this work were centered on the preparation and study of the Eu^{3+}-doped CNC, resulting in a film with iridescent characteristics, followed by the introduction of fibroin to obtain CNC-fibroin films. Photoluminescence studies have confirmed a better intensity emission from $Eu^{3+}$when excited by energy transfer from the cellulose structure. High values of pH in the film's preparation have provided more transparent and flexible materials, as well as an improvement in the emission parameters. Finally, further studies are been carried out for better knowledge about the coordination of Eu^{3+}ion at CNC structure, aiming to keep the cholesteric structure even after fibroin interaction.

Acknowledgements: Brazilian agency FAPESP (2019/18828-1), CNPq (303580/2021-6) and Universidade de São Paulo (USP) for financial support. Prof. Dr. Hernane S. Barud, from BioPolMat Group (UNIARA) for providing the bacterial cellulose membrane used.

References: [1] Vasconcelos, N. F., et al. Carbohydr. Polym., 155, 425-431, (2017), [2] Rockwood, D. N., et al. Nat. Protoc, 6(10), 1612-1631, (2011), [3] Guidetti, G., et al. Adv. Sustain. Syst., 5(6), (2021).

Base-catalyzed production of fatty acid oligomers from castor oil

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Estolides are oligomers composed of fatty acid (FA) monomers linked by mid-chain secondary ester bonds. These molecules can be synthetized from triacylglycerols (TGs), monoesters or free FAs and their size and properties depend on their degree of oligomerization and on further functionalization of the oligomer. The improved physico-chemical properties that the formation of estolides provide to natural fatty acid sources, such as vegetable oils, give to these special esters a wide range of applicability (e.g. base oils, lubricant additives, food emulsifiers and cosmetic formulations). Mineral acids, such as perchloric acid, and lipases are usually employed as catalysts for the production of estolides from unsaturation or oxygen-containing groups present in the aliphatic chains of FA bodies [1]. In this work, it was observed that the hydroxyl group present on the 12th carbon of the ricinoleic portion of castor oil (CO) acts as a transesterification agent when this oil is treated under chemical interesterification conditions (heating in the presence of a strong base as catalyst). Increases in viscosity in relation to the original oil (max. 146%) and HPLC and 13C NMR determinations confirmed the formation of acyl-glycerol estolides. Estolide numbers (EN) from 0.00 to 3.99 were obtained as a way to assess the degree of oligomerization for all samples studied. Due to the characteristics of the obtained material, its potential application as lubricants was investigated. In relation to the estolides reported in the literature and commercial lubricants with similar viscosities, the products obtained in this work presented advantages regarding the production process (in terms of number of reagents and reaction time) and most of the physical properties evaluated (pour point, flash point, ASTM color, viscosity index) attesting to the potential of industrial application of these materials.

References:[1] Y. Chen et al., JAOCS 97, 231 (2020).

Caracterização da celulose bacteriana obtida da membrana SCOBY

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A kombucha é uma bebida elaborada a partir do chá adoçado da Camellia sinensis fermentado com uma simbiose de bactérias e leveduras. Durante sua fermentação é formado um filme de celulose bacteriana (CB) na superfície do líquido chamado SCOBY(Symbiotic Culture of Bacteria and Yeasts) [1]. A celulose é o material orgânico mais abundante na natureza, e pode ser de origem vegetal, bacteriana ou animal, não diferindoem sua estrutura molecular e química. A celulose vegetal é a mais utilizada pela sua maiordisponibilidade e facilidade de obtenção, porém exige etapas intensas de purificação parasua utilização. Com isso, o objetivo deste trabalho é o reaproveitamento da CB do SCOBY,proveniente de resíduo industrial da produção de kombucha. Diferentes processos de obtenção, como por hidrólise ácida, mercerização e alcalinização tornam-se de grande valia para estudar possíveis aplicações do produto de acordo com suas propriedades. As análisesrealizadas nas CB extraídas foram composição centesimal e microscopia eletrônica de varredura (MEV). A quantidade de CB extraída foi acima de 90% para hidrólise ácida. Nasmicrografias observou-se que os tratamentos de mercerização e alcalinização foram insuficientes para separar as fibras, apresentando alto teor de carboidratos. As micrografiasdas amostras obtidas por hidrólise ácida mostraram comprimento de nanofibras. Assim, asnanofibras de celulose, de acordo com as análises físico-químicas realizadas e MEV, apresentam vantagens tecnológicas em relação à celulose vegetal [3]. References:[1]CHEN,C.;LIU,B.Y. Changes in major components of tea fungus metabolites during prolonged fermentation.[s.l.],p.834-839,2000.[2]JAYABALAN, R. et al. A Review on Kombucha Tea-Microbiology,Composition,Fermentation,Beneficial Effects,Toxicity,and Tea Fungus. [s. l.], v. 13, 2014.[3] LEE, K. et al. More Than Meets the Eye in Bacterial Cellulose :Biosynthesis,Bioprocessing,and Applications in Advanced Fiber Composites a.[s. l.],p. 10-32, 2014.

CELLULOSE-BASED HYBRID MATERIALS FOR DELAYED DRUG RELEASE IN WOUND DRESSINGMATERIALS

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Wound management is critically important in order to prevent wound infection and to facilitate the healing process. Traditional passive wound dressing material acts as a physical barrier to cover the wound, thereby lowering the chance of an infection. These wound dressing materials lack inherent antibacterial properties and/or wound healing properties. With the emergence of nanotechnology, methods have been explored in order to develop “active” wound dressing materials which stimulate tissue regrowth or provide an antibacterial effect. Nanocellulose has been of interest as a potential wound dressing material due to its beneficial properties (e.g. gas permeability, exudate removal), however it lacks antibacterial properties. Efforts have been made to combine antibiotics with nanocellulose materials, however these typically show low retention and rapid release of antibiotics. Effort has been made to control the release of antibiotics from nanocellulose materials by incorporating metal oxide nanoparticles. These particles are generally considered bio-compatible, with high affinity for antibiotics and pharmaceuticals. In this way a cellulose-based hybrid material was produced which permitted the release of the broad-spectrum antibiotic Tetracycline controlled by daylight.[1] A bacterial cellulose-based dressing material was modified with metal oxide nanoparticles to facilitate the delayed release of Tetracycline. Finally, a hybrid material was prepared for the controlled release of ampicillin[2]. Incorporating metal oxide nanoparticles into nanocellulose-based materials offers an approach to functional, biocompatible, hybrid materials capable of delaying drug release, which may be used as “active” wound dressing material.

References:

(1) Breijaert, T. C. et al. Carbohydrate Polymers 2022, 291, 119560. [2] Breijaert, T.C. et al. Mater. Adv. 2022, 3 (19), 7228-

Chemical-enzymatic conversion of ferulic acid intoepoxy resins: a renewable model to produce thermosetting materials

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Epoxy resins are thermosetting materials used in various applications such as adhesives, coatings and composites. Industrially, epoxy resins are produced based on bisphenol A, a petrochemical feedstock. This study aimed to develop a model of production of epoxy resin from ferulic acid, a renewable organic acid that can be obtained from plant biomass. Ferulic acid was dimerized by enzymatic oxidation using laccase [1], generating a dimer structurally analogous to bisphenol A. Ferulic acid consumption during reactions was measured by reverse-phase liquid chromatography. The reaction product mixture was subjected to an epoxidation reaction with epichlorohydrin to functionalize free phenolic hydroxyls with epoxy groups, with reaction efficiency monitored by differential UV spectroscopy. The epoxilated diglycidyl ether dimer was cross-linked with triethylenetetramine. The enzymatic oxidation reaction consumed all ferulic acid and formed several products, mainly the β-β dimer analogous to bisphenol A. The epoxidation reaction fully consumed the phenolic hydroxyls, confirming the functionalization with epoxy groups. The crosslinking of diglycidyl ether dimer with amine resulted in the formation of a glassy material with rigid characteristics, similar to commercial epoxy resins. The results of this study indicate the possibility of using a renewable feedstock for epoxy resin production as an alternative to the petrochemical route.

Acknowledges:
To FAPESP 2019/25867-3 and 2022/09174-0, and CAPES financial code 001.

Reference:

[1] Adelaku, O. E. Kudanga T., Parker A., Green I. R., Hill M. R. and Burton S. G., Journal of Molecular Catalysis B: Enzymatic vol. 74, pages 29-35 (2012).

Chemical functionalization of cellulose nanofibrils with hydrogen sulfate

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The disposal of untreated effluents in water bodies has caused serious impacts to aquatic biota, consequently making the water inappropriate for human consumption. In an attempt to mitigate the adverse effects, researchers are developing filter membranes from renewable sources [1,2]. The present work aims to develop filter membranes functionalizing cellulose nanofibrils (CNFs) for the removal of aquatic pollutant thus, the etherification reaction was carried out with a suspension of CNFs (3%) and the etherifying agent 2-aminoethyl hydrogen sulfate. Based on the 2^2 factorial design, four reactions were performed varying the temperature and reaction time. The chemical characterization was done using the Fourier transform infrared spectroscopic technique (FTIR), potentiometric titration, elemental analysis (AE), atomic force microscopy (AFM) and X-ray diffraction (XRD). The evidences that the chemical species under study was inserted in the CNFs are the increase in the intensity of the bands in the region of 2927 cm^{-1} and 2850 cm^{-1}, attributed to νasCH2 and νsCH2 and also the presence of other less intense bands attributed to the amino group; through potentiometric titration calculated the percentage of amino group (0.041%) and the degree of substitution (0.004); and 0.452% N (EA). To corroborate the initial results, the next steps will be to carry out pollutant removal tests.

[1] Pereira, A. R. Síntese e caracterização de um derivado de celulose aminado e sua utilização na remoção de Cu(II) e As(V) de soluções aquosos mono- e multicomponente. 149 f. Dissertação em Química - Universidade Federal de Ouro Preto, Ouro Preto/MG (2019).

[2] Genco, T. Amino cellulose sulfate: synthesis, characterization and physicochemical behavior. 117 f. Tese em Ciências Naturais - Faculdade de Química e Geociência da Universidade Friedrich Schiller, Jena - Alemanha (2013).

Chilean seaweed cellulose extraction

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The cultivation of macroalgae has stood out among the world's aquaculture sectors due to the transition to a low-carbon economy. In this sense, seaweed samples have the potential to be used as raw material for obtaining cellulose. Algae cellulose was first described in 1885. Since then, it has received great attention mainly due to its economic advantage. Cellulose extraction from the Chilean seaweed Ulva lactuca biomass was carried out following the methodology proposed by Paniz et al. [1]. After being harvested, the seaweed biomass was submitted to a pre-treatment of drying (35°C, 24h) and milling (knife mill). Then, it was forwarded to the desalination steps using deionized water at 110°C and removal of extractives using a Soxhlet system (toluene/ethanol at 7h). Finally, the sample was submitted to the following treatments: alkaline and bleaching using 5% NaOH and 0.1M $NaClO_2$solution, respectively. The sample was characterized using Solid-state nuclear magnetic resonance - 13C-CP-MAS. The cellulose extracted from the seaweed sample showed the typical signals of cellulose anhydroglucose carbon units at 105.7 ppm (C1), 89.8and 84.6 (C4), a large signal centered at 75.7 ppm (C2, C3 and C5) and 65.5 and 63.8 ppm(C6). Although peaks indicative of amorphous cellulose are more intense at C4 and C6 at 84.6 and 64 respectively, signals at 89.8 and 65.5 that correspond to crystalline cellulose were also detected. The cellulosic material was obtained without traces of impurities. Thus, the methodology proposed in this work was efficient in the extraction of cellulose from Ulva lactuca.

Acknowledgments:
CAPES (finance code 001)

References:

[1] Paniz, O.G., Pereira, C.M.P. et al. 2020 Cellulose. 27 113.

Composites of poly(butylene adipate-co-terephthalate) reinforced with cellulose fibers from coffee industry waste

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Coffee silverskin (CSS) is a thin tegument covering the coffee bean, and it is a by-product of coffee industry. Cellulose can be isolated from CSS to obtain cellulose fibers that could be incorporated into a polymer matrix, resulting in final reinforced products with high added value. Poly(butylene adipate-co-terephthalate) (PBAT) is a biodegradable polymer that can be used in the packaging sector, but its lower thermomechanical properties compared to other non-biodegradable polymers limits its use. In this work, alkali pretreatement was carried out on CSS fibers, followed by steam explosion. The cellulose fibers were incorporated into the PBAT matrix through melt fusion using a double-screw mini extruder (Minilab Rheomex). The properties of particles were evaluated by X-ray diffraction (XRD) and Fourier-transformed infrared spectroscopy (FTIR). Neat PBAT and its composites containing the same amount (2.5 wt.%) of different fibers (original CSS, pretreated CSS, pretreated, followed by a 2 bar-steam exploded-CSS and pretreated, followed by a 4 bar-steam exploded-CSS) were evaluated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The results of FTIR and XRD showed that alkali pretreatment and steam explosion had removed some of the amorphous components of CSS. The results of TGA and DSC showed same thermal stability (Tonset of around 375 ºC) and an increase in the crystallinity index (2.9%) of composites when compared to neat PBAT.Acknowledgments:The authors would like to acknowledge the financial support by CAPES and CNPq.References:[1] Murthy, P. S.; M. M. Naidu. Sustainable management of coffee industry by products and value addition - A review. Resources, Conservation Recycling, v. 66, p. 45-58, 2012. [2] Ferreira, F. V.; Cividanes, L. S.; Gouveia, R. F.; Lona, L. M. F. An Overview on Properties and Applications of Poly(butylene adipate-co-terephthalate)-PBAT Based Composites. Polymer Engineering and Science, v. 59, n. 2, 2019.

Development of sustainable filaments for 3D printingfrom PLA and modified Kraft lignin

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Despite being widely used in fused deposition modeling printing, poly(lactic acid) (PLA) has some mechanical limitations, such as low elastic modulus and excessive fragility, which restrict its use in functional applications [1]. In addition, its high cost compared to other types of filaments reduces its competitiveness compared to other available materials. In this sense, the inclusion of lignin can be an interesting strategy, since this biopolymer is an abundant component of biomass, obtained as a by-product of pulp processing and that does not compete with crops [2]. In this work, kraft lignin was chemically modified by esterification reaction with acetic anhydride and mixed with PLA using a twin-screw extruder to obtain a PLA/modified lignin blend. The acetylation reaction was evaluated combining the FTIR-ATR and DSC analysis, indicating both the successful modification of the kraft lignin and that the acetylated KLA will be suitable for incorporation into PLA. The modified lignin was incorporated into the PLA matrix, producing filaments of the PLA/modified lignin blends with a mass fraction of 70/30, respectively. DSC and DMA analysis (tanδ curve) suggested good compatibility between PLA/lignin, despite the high content of the latter. Combined analysis of rheology and DMA showed that PLA/modified lignin blends have desired characteristics for 3D printing, which are high G'' and h* in quasi-static conditions and low G'' and h* at high frequency. The final test of the filaments was performed on the 3D printer, in which it was possible to obtain printed samples.

Acknowledgements:

The authors acknowledge the financial support of the State of Sao Paulo Research Foundation (FAPESP - 2021/12726-2), as well as the Federal University of ABC for the support during the research process.

References:

[1] H. Dou, W. Ye, D. Zhang, Y. Cheng, Y. Tian. J. Mater. Eng. Perform. 31, 1925-1933 (2022).

[2] Gordobil, O., Delucis, R., Egüés, I. Labidi, J. Ind. Crops Prod. 72, 46-53 (2015).

EXTRACTION AND CHARACTERIZATION OF BACTERIAL CELLULOSE FROM SOLID BIOLOGICAL WASTE FROM KOMBUCHA PRODUCTION: APPLICATION OF NANOCELLULOSE IN THIN FILMSTO REPELLENT PROPERTIES

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The search for a healthier diet and the consumption of products with fewer preservatives and materials of synthetic origin is a current reality with a growth trend in the coming years. Kombucha can be a great alternative for a healthier diet and has many other benefits for human health.

The consumption growth and consequently the increase in the production of the beverage, the manufacturing chain inevitably generates rejects and residues, such as SCOBY disposal after successive productions due to the drop in productivity in terms of the capacity of cellulose synthesis and generation of acids through bacteria and fermentation capacity of yeasts.

The reuse of this waste can contribute economically and environmentally to the production process as a whole, for example, the use to obtain cellulose nanostructures in different segments, such as bio-sorbent cellulose film for effluent treatment, skin dressings or thin films for drug delivery.

The objective of this work is to obtain bacterial cellulose from homemade Kombucha waste through chemical bleaching treatment. Characterization of bacterial cellulose regarding its chemical composition, crystallinity index, thermal properties, and morphology in macro, micro, and nanostructured scales (nanocellulose). Obtaining bacterial cellulose micro and nanofibers through mechanical shear and characterization of bacterial cellulose and the possibilities of applying this material in superficial adhesives for repellent activity.

Fluorescent Carbon Dots Synthesized from Kraft Lignin via Hydrothermal Process

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Carbon dots (CDs) are photoluminescent nanomaterials with various technological applications, such as nanomedicine, sensors, biosensors, photocatalysis, organic solar cells and others [1-2]. However, their widespread use is hampered by the lack of potential raw materials with adequate [2]. This study aimed to produce CDs using Kraft lignin (KL), a co-product of the pulp and paper industry that is widely available in large scale. The CDs were synthesized via a hydrothermal process using KL and ethylenediamine as a dopant in distilled water. The produced material was a dark solution with a high dispersibility in water and a pH of 10.5. Under UV-A irradiation, the solution exhibited a blue-green color with a high degree of fluorescence. The UV-Vis analysis revealed a characteristic band at 277 nm, indicating the presence of the aromatic π-π* group, and a shoulder between 310-340 nm, suggesting n-π* of the C=O groups. The FTIR analysis confirmed the presence of functional groups of aromatic refusion of KL fragments. The zeta potential measurement indicated a surface charge of -31.7 mV at basic pH. The simple reaction process and the presence of negative charges indicate the potential application of these CDs for detecting heavy metal ions. This study demonstrates the possibility of producing carbon dots from KL, which could lead to a new direction for high value-added applications of this widely available co-product.

Acknowledgments:

FAPEAM, IFC/SFS, PIPE/UFPR, LCNano/UFPR, Embrapa Florestas.

References:

1. Li, Y., Ren, J., Sun, R. Wang, X. Fluorescent lignin carbon dots for reversible responses to high-valence metal ions and its bioapplications. J. Biomed. Nanotechnol. 14, 1543-1555 (2018).
2. Nguyen, T. N., Le, P. A. Phung, V. B. T. Facile green synthesis of carbon quantum dots and biomass-derived activated carbon from banana peels: synthesis and investigation. Biomass Convers. Biorefinery (2020)

Functionalized CNCs in homogeneous media as greener reactive filler in bio-based polyurethane foams: a journey towards sustainability

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In the last decades, the development of renewable materials and eco-friendly processes have gained much interest from researchers aiming to minimize the carbon footprint as wellas fossil fuels dependence. On this account, the replacement of the widespread petroleum-derived materials with greener and sustainable ones, had been widely performed by research communities. Nano-Crystalline Cellulose, CNCs is widely used as filler in polymer composites due to its high specific strength, tensile modulus, aspect ratio and in compliance with the principles of circular economy [1]. However, its hydrophilicity makes its dispersion complicated in hydrophobic polymeric matrices, resulting in aggregate structures, which compromise its reinforcing action. Here, the sytnthesis of modified CNCs via silanization in homogeneous media (N,N-dimethylacetamide/LiCl) and subsequently grafting with bio-based polyol is presented. Filler functionalization in homogeneous media allowed to graft up to 0.8 mmol of polyols per gram of CNCs. The polyol chemistry (i.e. molar masses and OH number) affects the functionalization effectiveness. On this account, bio-based composite Polyurethane foams, filled with 1-3 wt% of modified CNCs, were considered as case study and their chemico-physical, thermal, morphological and mechanical properties are investigated.

Acknowledgments:

This work has been financially supported by EU project-BIOMAT/H2020-NMBP-TO-IND-2018-2020. GA:953270.

References:

[1] Coccia, F., et al (2021). Chemically Functionalized Cellulose Nanocrystals as Reactive Filler in Bio-Based Polyurethane Foams. Polymers, 13(15), 2556.

Grafting for enhanced adsorption: Chemical modification of chitosan and nanocellulose for potential heavy metals, dyes, and pharmaceuticalremoval

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Abstract: Ensuring water quality and protecting aquatic life requires the removal of contaminants, and adsorption is a widely used, efficient, and cost-effective technique for water treatment, with many materials being studied for this purpose[1]. Nanocellulose and chitosan have demonstrated promising results as adsorbents for the removal of heavy metals, dyes, and pharmaceuticals due to their high surface area and unique properties. In a similar vein, the utilization of ethylenoxymethylenes and ethoxyacrylates has also shown potential for the adsorption of these contaminants[2]. These materials have been chemically modified through grafting to enhance their adsorption capacity and selectivity. This research area is still in its early stages, and further studies are needed to fully understandthe potential of these materials for water treatment applications. With the growing demandfor more sustainable and efficient solutions for contaminant removal, adsorbent materialshave become increasingly important for the protection of water and the environment as awhole[3].

Acknowledgments: We would like to thank CAPES for the financial support through the doctorate degree.

References

[1] HUANG, D. et al. Novel insight into adsorption and co-adsorption of heavy metal ions and an organic pollutant by magnetic graphene nanomaterials in water. Chemical Engineering Journal, v. 358, p. 1399-1409, fev. 2019.

[2] PEREIRA, R. V et al. A new route to produce chitosan derivatives: nucleophilic vinylic substitution with ethoxymethylenemalononitrile. Polymer International, v. 72, n. 3, p. 376-382, 22 mar. 2023.

[3] SARATALE, R. G. et al. Textile Industry Wastewaters as Major Sources of Environmental Contamination: Bioremediation Approaches for Its Degradation and Detoxification. In: Bioremediation of Industrial Waste for Environmental Safety. Singapore: Springer Singapore, 2020. p. 135-167.

Nanocellulose extraction from cultivated fibers inAmazon Forest

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Caesarweed ***(Urena lobata*** L.) and Jute (***Corchorus capsularis*** L.) are some of the fibers utilized on the Amazon Phoenix Project created by Instituto Nacional de Pesquisas da Amazonia (INPA). This project tries to build a synergetic sustainable network between human activities and the remaining natural ecosystems aiming for harmonious economic development [1].The use of these lignocellulosic fibers leads to an interesting field of research that works as renewable material, like cellulose nanocrystals (CNC) that can improve mechanical/thermal properties and biodegradability of matrices without generate a considerable increase in the cost of the process and making possible the development of value-added products, thus helping families who work with these fibers, the "Ribeirinhas" [2,3].

Keywords: Lignocellulosic fibers, Amazon forest, Cellulose nanocrystals, Acid hydrolysis, Caesarweed, Jute.

Acknowledgments
The authors to thank CAPES and Amazon Phoenix Project.

References

[1] Nobre, A.D. Projeto Fênix Amazônico desenvolve novos materiais compósitos com fibrasde árvores pioneiras da Amazônia, Blog. (2012) 1. http://fenixamazonico.blogspot.com/2012/05/projeto-fenix-amazonico-desenvolve.html (accessed December 6, 2020).

[2] Longaresi, R. H., de Menezes, A. J., Pereira-da-Silva, M. A., Baron, D. Mathias, S. L. The maize stem as a potential source of cellulose nanocrystal: Cellulose characterization from its phenological growth stage dependence. Ind. Crops Prod., 133, 232-240 (2019).

[3] Araújo, K. S., Pereira, H. S. Políticas públicas e as fibras naturais: a experiência recenteda cadeia produtiva da malva e juta amazônica, Rev. Ciências Agrar. - Amaz. J. Agric. Environ. Sci., 60, 60-69 (2017).

Nanocellulose from beer production waste: evaluation of the potential extraction by acid hydrolysis

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Industrial waste usage for technological applications has been gaining ground in different segments and applications. One of the prominent sectors is Material technology, where these residues can be used to obtain the raw material for obtaining fillers and reinforcement in thermoplastic polymeric materials. Cellulose has been used as a source of study and has several applications in materials, such as reinforcement, load, or chemical surface modifications. The nanocellulose, which is obtained from different chemical, mechanical and biological processes, can be in the form of cellulose nanofibrils (CNFs), which are present in crystalline and amorphous regions along it, and cellulose nanocrystals (CNCs) which reveal only the crystal region. The material in this work is malt bagasse, which is currently the waste generated in the beer industry by bulk and shows no alternative reuse and application in Material technology. Was determined the chemical composition of the fibers and applied a chemical bleaching process to remove components such as lignin and hemicellulose in preparation for the fibers for obtaining nanocellulose through acid hydrolysis. Hydrolysis was performed using H2SO4 at two concentrations, 44% (w/w) and 64% (w/w), at temperatures of 40°C and 50°C for the samples subjected to the acid solution at 44%, besides that, it was used at 40°C, 50°C and 60°C for samples with 64% solution. For thesamples, 44/40 and 44/50 were obtained CNFs and for the 64%, the result obtained wasCNCs. Natural and bleached fibers, along with nanocellulose, obtained through different concentrations of H2SO4 and temperature were characterized by Fourier Transform Infrared Spectroscopy (FTIR), Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM) and Field Emission Scanning Electron Microscopy (FESEM), X-Ray Diffraction (X-RD) and Thermogravimetry (TG).

Obtation and Characterization Kombucha Bacterial Cellulose: Biomedical Applications

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Kombucha (KC), known as an ancient beverage of Asian origin, is a promising source of bacterial cellulose (BC), which can be obtained through fermentation processes, such as green tea and sucrose [1]. The BC has stood out due to its unique properties, such as purity, crystallinity, biocompatibility and tensile strength, supporting commercial applications in different areas. This work aims to produce the bacterial cellulose membrane of Kombucha (MCBK), through a symbiotic consortium of yeasts and bacteria, cultivated by the fermentation of green tea and sucrose, kept in a light-free environment, varying the growth time and thus, determine the best obtaining conditions. MCBK were characterized by optical microscopy, SEM, AFM, XRD and FTIR, in order to enable the development of high added value products for biomedical applications, such as skin dressings for the treatment of skin lesions and burns [2,3].

Acknowledgments: The authors to thank CAPES for the financial support.

References

[1]Jaybalan, R; Malbala, R.V; Sathishkumar, M. Kombucha. Reference Module In Food Science, Elsevier, (2016), 1-8.

[2] Marestoni, L. D., Barud, H. S., Gomes, R. J., Catarino, R. P. F., Hata, N. N. Y., Ressutte, J. B., Spinosa, W. A Commercial and potential applications of bacterial cellulose in Brazil: ten years review. Polímeros, 30(4) (2020) 1-19.

[3] Dufresne, A. Preparation and Properties of Cellulose Nanomaterials, Paper and Biomaterials, 5 (3), (2020) 1-13.

Study on the use of nanocelluloses as reinforcement in composites with Poly(butylene adipate co- terephthalate) for potential application in biodegradable packaging

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Bioplastics are attracting great interest from the scientific community and industry, emphasizing biodegradable polymers. Poly(butylene adipate-co-terephthalate) (PBAT) is of greater commercial interest among bioplastics, as it is a biodegradable material obtained from bioderived raw materials. It is reported in the literature that nanocomposites reinforced with nanocellulose have properties superior to those of the original polymers, such as excellent mechanical and thermal properties. However, one of the limitations of using nanocellulose as reinforcement in polymers is the low affinity between its structure and the polymeric matrix due to the high number of hydroxyls on the surface. In this work, nanocelluloses with different morphologies were modified by acetylation in order to improve the interaction between the fillers and the polymer. PBAT composites were produced with the acetylated fillers, as well as with them without treatment, via mixing in the molten state in a mini twin-screw extruder at 160 ºC for 7 min. The effectiveness of acetylation was confirmed by infrared spectroscopy (FTIR). The characterization of PBAT through thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) showed that the processing did not significantly alter the polymer properties. Scanning electron microscopy (SEM) confirmed that the nanocelluloses showed good interaction and dispersion with the polymer matrix. The addition of fillers did not cause a decrease in thermal stability but promoted an increase in the hydrophobic character of the samples, confirmed by the contact angle with water. Compared to the pure polymer, the composites generally did not decrease their performance, which is important for the desired application.

Acknowledgments: CAPE UFRJ-IMA

References: [1] RAI, P.; MEHROTRA, S.; PRIYA, S.; GNANSOUNOU, E.; SHARMA, S. K. Bioresource Technology, v. 325, p. 124739 (2021).

Surucucumirá: a native Amazon tree with the potentialfor nano-reinforcement.

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Abstract: Surucucumira, a hapaxanthic tree, can be planted among others Amazon native trees providing shades and protection to slow growing species because of his fast growing, 10~20 meters of height and 20 centimeters of diameter in 2 years[1]. This specie's biomass chemical composition was inspected through TAPPI standards[2]. According to the analysis, lignin and alphacellulose accounted for around 39% and 31% of the total content. FTIR measurements showed the removal of lignin and hemicellulose from the raw fibers without breaking the glycosidic bond of the cellulose, corroborating with the SEM micrographs, that showed the released fibers (fibrillation) after the bleaching process. TGA showed the dependence of intermolecular and intramolecular relationships for a better, or worse, thermal stability of the samples. X-ray diffraction patterns reiterate type I cellulose for theraw/holo samples, and type II cellulose for the bleached ones. AFM showed the successfullyextraction of the cellulose nanocrystals (CNCs) and by calculating the aspect ratio and thespecific surface area it was shown that these CNCs might improve barrier properties onpolymers matrices[3].

Acknowledgments: We would like to thank CAPES for the financial support through the master degree, the Amazon Phoenix Project (Projeto Fênix Amazônico) for the fibers' donation and IFSC/USP for the AFM measurements.

References

[1] Rodrigues, W. A. Árvore hapaxanta na flora amazônica. Botânica 14 (1962).

[2] Longaresi, R. H., de Menezes, A. J., Pereira-da-Silva, M. A., Baron, D. Mathias, S. L. The maize stem as a potential source of cellulose nanocrystal: Cellulose characterization from its phenological growth stage dependence. Ind. Crops Prod. 133 (2019).

[3] Sanchez-Garcia, M. D., Gimenez, E. Lagaron, J. M. Morphology and barrier properties of solvent cast composites of thermoplastic biopolymers and purified cellulose fibers. Carbohydr. Polym. 71, 235-244 (2008).

Swelling properties and toxicity of babassu mesocarp (Orbignya speciosa) and bacterial cellulose hydrogels

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Polysaccharide hydrogels are hydrophilic compounds that make up a three-dimensional polymeric network capable of absorbing large amounts of water while maintaining its structural integrity. They are classified as superabsorbent when they can absorb water up to one hundred times their mass [1]. As bacterial cellulose has hydrophilic groups on its surface, it contributes to the water-retaining properties of the hydrogel. This work aimed to synthesize superabsorbent hydrogel from babassu mesocarp (HMB)| and bacterial cellulose (HCB). The samples were characterized by spectroscopy in the infrared region with Fourier transform, swelling test, and toxicity against Artemia salina. HMB showed characteristic vibration of the acrylamide monomer around 1670 cm-1, and the polyacrylamide bands masked the starch bands. For HCB, bands around 1335 and 1162 cm-1, corresponding to the asymmetric C-O-C stretching, are characteristic of cellulose II [2, 3]. The swelling capacity of the hydrogels was greater than 300 g/g. The ecotoxicity test against Artemia salina showed that after 24h, the number of live nauplii was greater than 80% in all concentrations used. After 48h, the number of live nauplii remained the same. The results presented showed the formation of the hydrogel and its non-toxicity, making it desirable for agriculture and drug adsorption applications.

T-Materials and Devices for Innovation in the Agribusiness

Additive manufactured microfluidic device for electrochemical detection of carbendazim in honey samples

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The use of pesticides is one of the primary means of protecting crops. However, this class of compounds can be highly toxic to the environment and humans [1]. Therefore, developing analytical devices for monitoring pesticides such as carbendazim in food sources is of paramount importance [1]. In this context, 3D-printed complete electrochemical platforms have gained increasing prominence in the literature, as it combines the qualities of electrochemical devices with the advantages and characteristics of 3D printing technology [2]. Thus, the present work presents the development of a paper-based microfluidic device coupled to an additive manufacturing platform and electrochemical sensors (produced from lab-made conductive filaments based on carbon black) for the sustainable detection of carbendazim in honey samples. All quantitative analyses were performed on a microfluidic paper attached to the 3D printed device with the electrochemical sensors attached and the drop volume was 30 μL for all analyses performed. For the application of the microfluidic device, the electrochemical sensors were previously electrochemically treated and characterized. Subsequently, the pH of the supporting electrolyte and the operational parameters of the square wave voltammetry technique were optimized. The microfluidic electrochemical device presented satisfactory results for the analysis of carbendazim, in the linear range from 0.5 to 40.0 μmol L-1 with a LOD of 0.09 μmol L-1. The recovery test performed in honey samples showed values ranging between 92.4 and 108.8%. According to the results, the proposed microfluidic device demonstrated the advantages of employing sustainable and renewable materials.

Acknowledgments:FAPESP (2017/21097-3, 2022/06145-0), CAPES (001; 88887.504861/2020-00) CNPq (380632/2023-3).

References[1] S. Singh and N. Singh, Environ. Chem. Lett. 14, 317-329 (2016). [2] J.S. Stefano and C. Kalinke. Anal. Chem. 94, 6417-6429 (2022).

Mineral nanoparticles as plant stress mitigation and growth promotion agents

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Life has emerged and developed in constant presence of mineral nanoparticles (MP). Dissolution of tectonic silicate minerals, major components of the earth crust, leads continuously to formation of sand minerals, originally - in the form of nanoparticles. The latter are undergoing aggregation and growth forming micro and macro particles. Major sand minerals, originating from these reactions are common sand, SiO_2, iron oxides FeO_x, aluminium oxide-hydroxides, manganese oxides, MnO_x, and titanium dioxide, TiO_2. Other major fraction of sand is composed of calcium and magnesium carbonates. Beneficial effects of flooding rivers on crop growth have been well recognized in human history. The are usually explained to be partly due to the presence of MPs in the sediment, through their ability to adsorb and release nutrients and microelements.

In our studies we have demonstrated that in particular TiO_2MP can be generated by plants themselves on interaction of plant root exudates with soil minerals [1]. The beneficial role of MP was shown to be in particular due to their ability to enhance bacterial adhesion to the roots and formation of large clusters of plant growth promoting rhizobacteria (PGPR) [2], resulting in beneficial effects via plant stress mitigation. Another important mechanism of MP nanomaterials is nanozyme effect that can be used to regulate the concentrations of Reactive Oxyges Species (ROS) in plant tissues – a feature typical of iron oxides and ferrites [3]. Controlled oxidation of key amino acids mediated by MP as nanozymes can lead to improved synthesis of plant hormons improving biomass production [4]. These properties make MP attractive as additives in seed treatment for improved plant survival and growth.

References

[1] G.A. Seisenbaeva et al. Nanoscale 2013, 5, 3330-3336.
[2] N.G.M. Palmqvist et al., Sci Rep 2015, 5, 10146.
[3] N.G.M. Palmqvist et al., Nanoscale Res Lett 2017, 12, 631.
[4] V.G. Kessler et al. (to be published).

Raman Spectroscopy, FT-IR, TGA-DTA and DFT calculation on the complex of Monoethanolamine with the transition metal Cu (II).

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The science of coordination compounds is very important for areas such as nanotechnology, biomedicine, agrochemicals, new materials, and others. In the agrochemical area, a molecule of biological interest, by being coordinated with metal, can increase the efficiency of the active principle and reduce its toxicity. Currently, copper (II) is used in a wide range of industrial processes, with remarkable biological importance in different types of organisms, as in plant photosynthesis and as an active center of certain proteins and enzymes. Studies have also shown that Cu (II) compounds coordinated to monoethanolamine (MEA) have antifungal action, improving the defense of plants against certain pests, such as in the formulation of wood preservatives, and have moderate antimicrobial activity too. The vibrational, electrochemical, and thermal properties of the monoethanolamine (MEA) complex were investigated by combining infrared spectroscopy and Raman at room temperature, assisted by density functional theory (DFT) calculations. Calculations on B3LYP and LANL2DZ (which contain ECP information) led to the identification of the minimums on the potential energy surfaces of the molecules. These calculations predicted the most stable conformer was predicted in relation to the dihedral angle H9-C4-N7-Cu1. A more precise understanding of the fundamental vibrational modes is obtained by comparing experimental and theoretical vibrational spectra, which also improves the interpretation of experimental Raman and infrared spectra. The vibrational attribution given in terms of potential energy distribution analysis (PED) leads us to a more comprehensive interpretation of the vibrational spectra of the investigated materials and electronic properties. Time-dependent DFT was used to derive HOMO and LUMO (TD-DFT). The gap energy, which was determined by comparing the HOMO and LUMO orbitals, was calculated at 0.786 eV

Antibiofilm activity and synergism of essential oils and antibiotics (oxacillin) encapsulated in electrospun fibers of cellulose acetate

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The World Health Organization (WHO) has warned of the increase in contamination by food infected with microorganisms that affect about 10% of the population, with a high death rate. These data lead to great concern, as revealed by the increase in the use of chemical and synthetic products to minimize losses in processed and fresh foods, which contributes to bacterial resistance mechanisms against conventional antibiotics [1]. Thus, the use of alternative strategies to avoid/ minimize antibiotics in high concentrations is necessary to control the bacterial population. The objective of this work was to evaluate the in vitro antibacterial activity of electrospinning fibers of cellulose acetate associated with essential oils of clove, cinnamon and eucalyptus and their combinations (clove + cinnamon, clove + eucalyptus and cinnamon + eucalyptus) in addition to the association of essential oils with oxacillin. The response of the electrospun systems was evaluated using agar diffusion assays, the kinetics of time of death and the antibiofilm response of the modified fibers. The best experimental systems were fibers with clove essential oil in combination with cinnamon essential oil, which allowed the complete elimination of S. aureus from an initial concentration of 108 CFU/mL after 3 hours of treatment, showing even better results for inhibition of biofilm with a reduction of 47% and 65% for fibers with clove and with clove + cinnamon, respectively, confirming superior results for the use of essential oils reported in a new experimental system, which allows the high surface area of electrospinning fibers (in association with porosity) and the diffusion of oil along the fibers for the continuous action of the active compounds to be extended and applied in products such as wet wipes for bacterial removal.

References[1] E. J.,Dierings de Souza, D. H. Kringel, A. R. Guerra Dias, E. da Rosa Zavareze, Carbohydr. Polym. 265, 118068 (2021).

Design and fabrication of hybrid supraparticles with potential application in agriculture

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Current agricultural practices are incipient and will not be able to meet the population's demand for food. In an attempt to change this scenario, agrochemicals are used in an exacerbated way. However, studies show that only about 0.1 to 10% of agrochemicals reach the target plant, with the rest being degraded or bioaccumulated in soil and groundwater [1]. Nanotechnology based on developing controlled release nanocarriers has been a promising agriculture technology since it contributes to reducing environmental impacts. However, challenges have arisen related to toxicological aspects, safety, as well as understanding the behavior and mechanism of action of nanocarriers in complex biological matrices. Therefore, advances in science have allowed the combining of individual nanoparticles into more complex and advantageous systems, also called supraparticles (SPs) [2]. In this study, a superhydrophobic surface was prepared to synthe SPs using the evaporation-directed synthesis method. Thus, hybrid SPs were synthed from zinc oxide nanoparticles (ZnO NPs), poly-epsilon-caprolactone nanocarriers containing magnetic nanoparticles (Fe_3O_4@OA NPs), and herbicide atrazine (PCL:ATZ:NPs Fe_3O_4@OA). Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) analysis evidenced the presence of NPs inside the SP with spherical morphology. Moreover, Zn and atrazine were slowly released from the SPs. This innovative system opens up prospects for a new hybrid agrochemical delivery system. Acknowledgments: The authors acknowledge the FAPESP (#2022/03219-2 and #23/00541-3), CNPq (#427498/2018-0, #161360/2021-1), and CAPES for the financial support. References:

[1] Zhao, X., Cui, H., Wang, Y., Sun, C., Cui, B., Zeng, Z. Journal of Agricultural and Food Chemistry, vol. 66, page 6504-6512, 2017.

[2]Mattos, B.D., Greca, L.G., Tardy, B.L., Magalhães, W.L.E., Rojas, O.J. Small, vol.14, 2018.

Development of a bioactive membrane based on a functionalized composite with nanoparticles of Simonkollite (SK) and Simonkollite silver (SK-Ag)

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Active packaging has become a promising source of research since a large part of the food has been infected by infectious agents such as bacteria, fungi and viruses still on the shelf. Simonkollite ($Zn_5(OH)_8Cl_2H_2O$) also known as zinc hydroxide chloride compound monohydrate, naturally found in German mines, is a derivative of Zinc Oxide corrosion with potential microbial activity [1]. Staphylococcus aureus is a bacterium that can cause diseases, infections, pneumonia, meningitis, and others, it was used in this work to testing that effectivity of biofilms. The objective of this work were the development of a bioactive film based on chitosan ⁄silica composite functionalized with nanoparticles of Simonkolleite (SK) and Simonkolleite silver (SK-Ag). In order to develop of the biofilm, tests were performed evaluating the concentration composite (0.75%, 1% and 1.5% m/m) and nanoparticles (1%, 2% and 5% v/v). These materials were characterizated by thermogravimetric analysis (TG), FTIR, scanning electron microscopy (SEM) and X-Ray Spectrometry (EDX). In order to evaluate the antibactericidal activity of the Composite SK5% and composite SK-Ag (5%) films, a test was performed to determine the antibactericidalactivity where the Staphylococcus aureus ATCC 25923 (Laborclin) strain obtained the antibacterial ratio of the control test groups that was performed by direct contact method,respectively after 1 hour with the standardized inoculum. Despite of numerical values of allnanocomposites showed antibacterial activity against S. aureus, the SK-Ag was 10% moreeffective due to the additional effect of Ag. In view of the above, the evaluated films havethe potential to be use as active packaging.

Acknowledgements:
The authors acknowledge support fromFederal University of Alagoas, LEMAN, FAPEAL and CNPQ.

References:

[1] SILVA, A. C. A.; SILVA, M. J. B.; ROCHA, A. A.; COSTA, M. P. C.; MARINHO, J. Z.; DANTAS, N. O. Materials chemistry and physics, v. 266, 124548, 2021.

DEVELOPMENT OF HYBRID POLYMERS FOR CONTROLLED RELEASE OF SEMIOCHEMICALS

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During fruit production, it is important to overcome the enormous challenge of controlling pests such as the fruit fly [1]. Therefore, this work developed an adsorbent material based on silica precursor and biopolymer (cotton línter purified), capable of controlling the releaseof semiochemicals [2]. This material was characterized by several techniques such as FTIR,SEM, XRD, EDX, BET and tested for the release of ethyl octanoate, which is one of the compounds present in the pheromonal mixture of the fruit fly. The material had an area of 340 m2/g and significant results in bioassays for the attractiveness of mated females of the Anastrepha Obliqua species, with an adsorption capacity of 213.5 mg/g and a release rate of 1,087 mg/h. The decomposition profile and thermal stability showed that the hybrid will resist the conditions to which it will be submitted in the field. With this, it is intended to produce a prototype of attraction and subsequent elimination that will be used in integrated management programs contributing to the population control of pests.

Acknowledgment:
Federal University of Alagoas, LEMAN, CNPq, FAPEAL.

References:
[1] DE AQUINO, NATHALY; DO NASCIMENTO, RUTH, JOURNAL OF CHEMICAL ECOLOGY, vol. 47, p. 167-174, 2021.
[2] OLIVEIRA, YGOR; ABREU, FABIANE, QUIMICA NOVA vol. 44, p. 425-431, 2021

Easy production of corn zein micro- and nanospheres with the using of Ultrasound

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Zein is a protein of the groups of cereal prolamins, in the case of this protein, found in greater quantity in the endosperms of corn grains, reaching around 8% in this region along with starch. In order to encapsulate the starch globules with the various types of zein, which together with carotenoids in the cross-section of the grains forms a vitreous material with zein and starch that is too hard to withstand physical damage in transports and insect attack. With the development of the extraction process by the group of crude zein from corn gluten bran, this work aims to prepare micro- and nanospheres of zein from this crude zein with the aid of ultrasound to verify if there is a reduction in at a temperature of 25°C. The results of scanning electron microscopy of the zein spheres showed that the reduced about 10% with s between 90 to 550nm. This reduction must be because of the ultrasound vibrations of the drops that can improved with the use of capillary of smaller diameter getting with higher concentration of the nanospheres close to 200nm. In these nanospheres, we want encapsulate jambolan extract to treat the symptoms of herpes zoster on the skin in the body of people affected by this infection, or encapsulate bioinseticides or biofertilizers to control delivery.

Acknowledgements:
Ingredion Inc. for supply of CGM60.

References:

[1] Rodriguez-Félix, F. Food Science and Biotechnology, v.29, p.619-629 (2020).

Electrolyte-gated Transistors Using Bovine Milk As Electrolyte

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There is a growing demand for the use of biocompatible and eco-friendly materials to compose parts of electrolyte-gated transistors (EGTs), since this class of devices can be applied in situations as e.g., artificial synaptic devices, basic building blocks in bioelectronics or biosensors. EGTs can operate as electrolyte-gated field effect transistors (EGOFET), being modulated by the gate field effect, or as organic electrochemical transistors (OECT), being modulated by the ionic current. In this work, it is depicted a sequence of measurements performed on EGTs with the following structure: gold electrodes with a gate in-plane, poly(3-hexylthiophene-2,5diyl) (P3HT) as the channel and commercial gelatin foil hydrated with deionized water or bovine milk as electrolyte layer. Gelatin is a natural protein from animal being also a bio-compatible and low-cost material as well as bovine milk. The electrical characterization was performed based on the typical transistor’s curves: transfer and output curves, added by a simple IxV curve [1]. Transistor measurements using only gelatin hydrated with deionized water was considered as reference measurements (base-line signal). When water is replaced by milk, it was observed that the EGOFET to OECT transition regime has been changed, being reduced by 0.1V, and that the leakage current through the electrolyte was increased. Even so, it was possible to carry out a sequence of measurements showing that the transistor structure with bovine milk operates in EGOFET and OECT modes depicting the typical transfer and output curves profile. These data open opportunities for future applications as a platform for sensing adulterated milk.

Acknowledgments:

The authors thank to Group of Organic Optoelectronic Devices - UFPR for stuff and technical support.

References:

[1] Elton A. de Moura et al. J. Appl. Phys. 129, 154502 (2021).

Elucidating the cumulative cytotoxicity of pesticides employed in sugarcane agriculture on human lung carcinoma cells

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Brazilian agriculture''s national and global significance coexists with the widespread use of pesticides. The production of sugarcane has established Brazil as a prominent player in the agricultural industry, with the state of São Paulo leading the country''s production. Furthermore, the mesoregion of Assis is noteworthy for exceeding the state''s average production. In this scenario, simazine (SI) and methyl parathion (PM) have been extensively consumed and they are among the 27 pesticides detected in river waters that supply the mesoregion of Assis. Although these pesticides are regulated by Law, their cytotoxic effects have been poorly explored on human health. Due to inhalation, the lung is a very vulnerable organ to such toxins and can be considered a target for the cumulative cytotoxicity of these pesticides. In this study, we have elucidated the cumulative cytotoxicity of the pesticides SI, PM, and their cocktail (mixture of these pesticides) on in vitro culture of cells derived from human lung carcinoma (A549). The cytotoxicity assays were performed by incubating A549 cells with two different concentrations of the pesticides and their cocktail (10-5 mol/L and concentrations regulated by Law) for 24, 48, 72 and 96 h. The results showed a reduction in cell viability with an increase in the incubation period, indicating a cumulative cytotoxicity of these pesticides over time. Moreover, higher concentrations of the pesticides exhibited a stronger cumulative cytotoxic effect of the cocktail, indicating a synergistic cytotoxicity. Thus, despite being released for use in sugarcane agriculture at concentrations regulated by Law, preliminary in vitro studies have shown the cumulative cytotoxic effects of these pesticides on lung cells. We shall investigate the morphological modifications and membrane permeabilization to obtain more evidence and a better understanding of the cumulative cytotoxicity of these pesticides. Acknowledgements: FAPESP, Capes and CNPq.

Enhancing the quality and microbiological control in controlled environment agriculture (CEA): Harnessingthe Ultraviolet Light (UV-C) treatment in hydroponic systems.

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Plant microorganisms have the potential to benefit plant growth by producing natural antibiotics, but they can also spread diseases that impede growth due to competition for nutrients. Plant management and environmental contamination can lead to various types of contamination, causing infections and hindering plant growth (1). In hydroponic systems, fluid recirculation can spread microorganisms through water and infect crops (2) making it necessary to adopt decontamination methods. This study explores the use of UV-C light for microbiological control in hydroponic lettuce production in a controlled environment agriculture (CEA), examining the effects of its application. We studied the effects of UV-C light on the growth and biochemistry of hydroponic lettuce, including height growth time, pigment content, phenolic compounds, antioxidant capacity, and mineral levels. Samples exposed to UV-C light in the hydroponic fluid, were compared to control groups without UV-C application, under 18 and 20-hour photoperiods. Applying the UV-C decontamination system to nutrient solution promoted faster plant growth while maintaining or even increasing nutritional values compared to non-treated groups. That technique has significant potential for accelerating vegetable production, reducing production time, and enhancing the absorption and content of beneficial compounds.

(1) Dankwa, A. S., Machado, R. M. Perry, J. J. Sources of food contamination in a closed hydroponic system. Lett. Appl. Microbiol. 70, 55-62 (2020).

(2) Olle, M.; Virsˇile,̇ A. The effects of light-emitting diode lighting on greenhouse plant growth and quality. Agricultural and food science, Helsi´nquia, v. 22, p. 223-234, 2013

The authors acknowledge the financial support provided by FAPESP Proc. № 13/07276-1 and INCT Proc. № 573587/2008-6). and Lara Perez S. acknowledges CAPES for doctorate grant 88887.663420/2022-00. All the authors thank C4 Cientifica for all technical support.

Fabrication of hybrid beads for controlled release ofzinc in the soil

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Zinc deficiency is the most ubiquitous micronutrient deficiency problem in world crops. Hence, zinc fertilizers are used to prevent Zn deficiency and in the biofortification of several cultures worldwide [1]. However, novel technologies are emerging to create eco-friendly formulations of zinc that are cost-effective and less toxic to the environment. In this study, hybrid beads were fabricated using a blend of biopolymers such as alginate, cellulose, and lignin to encapsulate zinc nanoparticles (Zn NPs) to produce a Zn-controlled release system in the soil. The beads containing Zn-nanoparticles were prepared by the co-precipitation method and characterized by several analytical techniques [2]. Optical microscopy (OM) and scanning electron microscopy (SEM) analysis of hybrid beads showed spherical morphology and distribution of c.a. 3 mm. X-ray scattering spectroscopy (EDS) confirmed the presence of Zn nanoparticles uniformly distributed inside the beads with energy peaks in the range of 8.65 keV for Zn. Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) indicated interactions between polymers and Zn NPs. In vitro release assays showed that Zn NPs-encapsulated in biodegradable beads can release zinc ions to the environment in a controlled way. Therefore, our system has the potential to change the bioavailability of zinc in the soil and may contribute to better absorption of micronutrients by plants.

Acknowledgments: The authors acknowledge the FAPESP (#2022/03219-2 and #23/00541-3), CNPq (427498/2018-0), and CAPES for the financial support.

References:

[1] Praharaj, S., Skalicky, M., Maitra, S., Bhadra, P., Shankar, T., Brestic, M. Hossain, A. Molecules, 26(12), 3509,2021

[2] Mandal, S., Kumar, S. S., Krishnamoorthy, B., Basu, S. K. (2010). Brazilian journal of pharmaceutical sciences, 46, 785-793, 2010

Fabrics functionalized with MOF-808 for glyphosate adsorption

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Glyphosate (GLY) is a herbicide organophosphate, active ingredient of the Roundup® commercial formulation and present in hundreds of other agricultural pesticides. This organophosphate is a neurotoxic agent, also considered as potentially carcinogenic by the Agency International Committee for Research on Cancer (IARC).1 Faced with occupational exposure to GLY, it is necessary to develop filters that are efficient in its adsorption, aiming at the production of masks and personal protective clothing that avoid intoxication of farmers, especially chronic intoxication. Zirconium-based metalorganic frameworks have been showing excellent performance in the adsorption of GLY. In this work, MOF-808 was synthesized in-situ (FI) and ex-situ (FE) on cellulose fabric substrate (cotton fabric), subsequently tested in Roundup® brand commercial GLY adsorption, both in solution immersion and in aerosol tests. The presence of MOF-808 in tissue was confirmed by DRX and SEM. In immersion adsorption tests, the fibers removed 99.8% (FI) and 99.6% (FE) of GLY in solution after 3 hours of contact, while the unmodified fiber (FNM) achieved 10.2% removal. The Langmuir model showed the best fit to the adsorption isotherm obtained, reaching the maximum adsorption capacities (qmax) of 10.8 mg g-1 (FI) and 6.38 mg g-1 (FE). In aerosol tests, the composites showed increased efficiency by 73% (FI) and 87% (FE) compared to the performance of FNM, indicating that the presence of MOF-808 in the fabric increases its efficiency in adsorbing glyphosate. These results demonstrate the potential of cellulose-MOF composites in the development of personal protective equipments for manual application of GLY. Acknowledgements: To LabPetro and LUCCAR (UFES), BSTR (UFPE) and LQSN (UFF) for technical support. References: [1] Ferreira, C., Durán, R. & Faro, L. R. F. Toxic Effects of Glyphosate on the Nervous System: A Systematic Review. Int. J. Mol. Sci. 23, (2022).

Germination responses of Oryza sativa L. seeds by the application of sophorolipids derived from Starmerella bombicola

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In order to foster more sustainable agricultural practices, biotechnology may play prominent role in the transition of vegetal monocultures to a more diverse agriculture aiming to preserve and assure biodiversity variability. In this context, biosurfactants (BSs) and specifically, sophorolipids (SLs) derived from yeasts such as Starmerella bombicola can affect the dynamics of cellular membranes which in turn may cause amendments in their permeability, fluidity as well as hydrophobicity [1]. These amendments may stimulate the immune system of plants and confer resistance to abiotic and biotic stresses [2]. In this sense, this study has aimed to evaluate the germination responses of Oryza sativa L. seeds by the employment of SLs treatment. Regarding the results, SLs in lower concentration (0,15 g/L) conferred notable improvement to the vigor of germination, total seeds germinated, the size and number of roots, and the dry total biomass compared to the control (H20). Thus, it is expected to contribute to the development of eco-friendlier bioproducts to promote sustainable agriculture worldwide.

Acknowledgments:This work is part of the project “INCT Yeasts: Biodiversity, preservation and biotechnologicalinnovation”, funded by Conselho Nacional de Desenvolvimento Científico e Tecnológico(CNPq), Brasília, Brazil, grant #406564/2022-1.

References:[1] E. Kaczorek, A. Pacholak, A. Zdarta, W. Smułek. Colloids and Interfaces 2(3),35 (2018).[2] R. Schellenberger, M. Touchard, C. Clément, F. Baillieul, S. Cordelier, J. Crouzet, S. Dorey 2019. Molecular plant pathology 20(11), 1602-1616 (2019)

Layered double hydroxide as nanocarrier of Tryptophanfor the development of novel agro-inputs

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The application of free amino acids has multiple functions as biostimulants of plant growth and development. Particularly, the use of Tryptophan (Trp) has positive effects on crop growth and productivity, as well as mitigating the negative effect abiotic stress [1]. Nevertheless, tryptophan (Trp) is an aromatic chemical compound that is susceptible to photooxidation and degradation upon exposure to UV light, which give it limitations when it comes to ensuring its effectiveness on a productive scale [2]. Layered double hydroxides (LDH) belong to the class of anionic clays whose structure is based on layers similar to brucite (Mg(OH) [3]. Our working hypothesis is based on the fact that LDH functionalized with Trp (LDH-Trp) have high potential as a novel active compound for application purposes, since the functionalized nanoclays confer stability to the active principle. In the present work, an HDL of Mg-Al was synthesized by co-precipitation and subsequently modified by ion exchange to tailor it for use as a Trp nano-carrier. The developed nanocarrier was characterized by Fourier-transform infrared spectroscopy (FTIR), which demonstrated the successful incorporation of Trp into the LDH matrix. Subsequently, the release profiles of Trp were determined. The biological functionality of LDH-Trp was tested in a bioassay in Arabidopsis thaliana plant model system. The primary root growth of Arabidopsis seedlings is strongly inhibited by high concentrations of free Trp. Arabidopsis seedlings cultivated in LDH-Trp exhibited similar root growth than free Trp. Our findings suggest a promising scenario for employing LDH as a nanocarrier of Trp in the development of novel agro-inputs.

Acknowledgment: CONICET, UNMdP, Agencia I + D + i.

References:

[1] Mustafa, A., Imran, M., Ashraf, M., and Mahmood, K.J.P. (2018). Pedosphere; 28, 16-34.
[2] Zhang, K., Fei, W., Ji, J., and Yang, Y.J.W. (2021). Water; 13, 2368.
[3] Bi, X., Zhang, H., and Dou, L. (2014). J Pharmaceutic 6, 298-332.

MOF-808 on Roundup® commercial glyphosate adsorption

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Glyphosate (GLY) is an organophosphate herbicide, active compound of the commercial formulation Roundup® and hundreds of other agricultural pesticides, and is currently the most sold herbicide in the world.[1] Metal-organic frameworks (MOFs) have been investigatedfor adsorption of GLY due to their porosity and availability of active sites, highlighting in this application zirconium-based MOFs.[2] In this work, the performance and kinetics of adsorption of glyphosate from the dilution of Roundup®, by MOF-808, until then not investigated for this application, were investigated. This MOF was synthesized by two different routes, one in organic solvent (MOF-808-DMF) and the other in aqueous medium (MOF-808-H_2O). The Langmuir model showed a better fit to the adsorption isotherm obtained by both MOFs, reaching the maximum adsorption capacities (q_{max}) of 273.2 mg g^{-1} and 277.0 mg g^{-1} for MOF-808-DMF and MOF-808-H_2O, respectively. Although they have approximately the same qmax value, MOF-808-DMF has better kinetic performance compared to MOF-808-H_2O, adsorbing 70% of the GLY in solution in just 10 minutes. This performance is possibly associated with its smaller particle size (277,0 nm), which increases the availability of external adsorption sites compared to MOF-808-H_2O (596.5 nm), which are the first to be reached after the intrafilm diffusion of GLY. These results confirm the potential application of MOF-808 in the adsorption of GLY and demonstrate the importance of textural characteristics in the adsorption performance of MOF-type materials.

Acknowledgements: To LabPetro and LUCCAR (UFES), BSTR (UFPE) and LQSN (UFF) for technical support.

References: [1] Ferreira, C., Durán, R. Faro, L. R. F. Toxic Effects of Glyphosate on theNervous System: A Systematic Review. Int. J. Mol. Sci. 23, (2022). [2] Zhu, X. et al. Effective adsorption and enhanced removal of organophosphoruspesticides from aqueous solution by Zr-Based MOFs. ACS Appl. Mater. Interfaces 7, 223-231 (2015)

Nanoclay-Based Bioproducts for Plant Protection

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The indiscriminate use of fertilizers and pesticides has been linked to environmental pollution, the emergence of agricultural pathogens, and a loss of biodiversity. To address these issues, current efforts are focused on researching eco-friendly alternatives for protecting plants from stress. The aim of our project is to use nanoclays such as bentonite (Bent) and anionic clays such as layered double hydroxides (LDHs) to deliver active compounds such as Salicylic Acid (SA), a phytohormone that provides stress protection in plants. Previously, we used Bent as a delivery matrix for SA (Bent-SA), demonstrating that foliar application induces innate defense mechanisms in plants and provides protection against biotic stress [1]. However, the incorporation and sustained release of SA was not optimal in this matrix. Therefore, in this study, we aimed to use alternative matrices for delivery: LDHs (LDH-SA) and Bentonite previously functionalized with Chitosan (Ch), a polysaccharide with antimicrobial activity and plant growth-promoting properties (Bent-Ch-SA). The developed nanocomposites were characterized by Fourier-transform infrared spectroscopy (FTIR), demonstrating the incorporation of SA in both matrices used. Using UV-Vis spectroscopy, we determined the loading efficiency of SA and the in vitro release profiles in aqueous solutions. The results indicate that Bent-Ch-SA retains a maximum of 5,42 % of SA and releases a maximum of 13,5% after 30 extractions of the resuspension medium, while HDL-SA retains 16,4% and releases 40,1%. Western blot assays revealed that defense markers were accumulated at 48 h upon application in tomato seedlings. Our findings indicated that nanoclay systems are promising bioactive carriers to improve growth and stress protection in plants.

Acknowledgements: UNMdP (EXA 1046/21), CONICET y Agencia I+D+i (PICT 2019 3002; PICT Start up 2020 0023)

References: [1] A.Y. Mansilla et al. Eur J Plant Pathol 158, 211-222 (2020).

Novel one-step polymeric nanogels based on polyetheramine-bis-epoxide as micronutrientnanocarriers for agriculture.

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Developments in the area of nanotechnology have enabled important advances in the area of agriculture. In this regard, nanogels have very interesting properties, such as good biocompatibility, high porosity and high loading capacity for micronutrients used in agriculture. These nanogels can be prepared from a simple one-step method by heterogeneous polymerisation of monomers [1,2]. These nanogels have been observed to exhibit sustained release of micronutrients over time [3]. In this work, we evaluated the formation of nanogels based on polyetheramine (PPO) and bis-epoxide (BisE). An Fe salt was used as a micronutrient to load into the nanogels. The nanogels were physicochemically characterised by DLS, zeta potential (ZP), TEM, SEM and biological assays were performed to evaluate their cytotoxicity. It was observed by TEM and DLS that these nanogels have a size between 200 and 500 nm depending on their composition, and that the size increases as a function of the micronutrient concentration. The surface charge was measured by ZP. At basic pH, the nanogels show a negative surface charge and for pH below 7, their surface charge is neutral or slightly positive. During cytotoxicity tests, it was observed that the nanogels have high cell viability and are viable for use in agriculture.

Acknowledgements: FAPESP (grants #2021/06552-1, #2022/06507-9, #2022/13408-7 and EMU #2021/14619-9), CAPES (Finance Code 001), and CNPq (grant #307696/2021-9).

References:

[1] S. Tang, Z. Shi, Y. Cao, W. He, J. Mater. Chem. B. 1 (2013) 1628-1634.

[2] C. Qin, Y. Lv, C. Xu, J. Li, L. Yin, W. He, Y. Lv, Y. Zhao, C. Xu, Z. Jin, C. Qin, L. Yin, S. Tang, L. Huang, R.J. Daniels-Mulholland, E. Dlugosz, E.A. Morin, S. Lenaghan, W. He, N. Kapate, C.W. Shields, S. Mitragotri, Int. J. Pharm. 484 (2020) 15-40.

[3] B. Ndaba, A. Roopnarain, B. Vatsha, S. Marx, M. Maaza, ACS Agric. Sci. Technol. 2 (2022) 1218-1229.

Performance analysis of natural fiber and glass powderas reinforcement and supplementary cementicious material in mortar

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The use of waste and the reduction of cement consumption is a growing demand in the civil construction industry [1]. This study aims at the production and characterization of mortars with glass powder as a partial substitute for cement and babassu coconut fiber (FT) as an additional reinforcement, in order to contribute in a sustainable way to a better disposal of this waste. The mixtures were prepared with a ratio of 1:3 cement/sand (in mass) and 0.7 water/cement. Cubic samples were prepared and named 0%/FT (0% glass powder and 0.5% FT) and 10%/FT (10% glass powder and 0.5% FT). By XRD (X-Ray Diffraction) the FT chemically treated with NaOH, showed an increase in crystallinity due to the removal of non-cellulosic (amorphous) components, which may contribute to better adherence of the fibers/mortar. FRX (X-ray Fluorescence) confirms the pozzolanic characteristic ($SiO_2+Al_2O_3+Fe_2O_3$ >50%) of the glass powder. For mortars, 0%/FT and 10%/FT, the mass consistency indices of respectively 27.3 ± 0.6 and 28.6 ± 0.3 cm were obtained. Thelow hydrophilic character, low FT content and absence of water absorption by the glasspowder, maintains the water/cement ratio effectively present in the mortar, so that the small variation between the results is influenced by the greater packing (density) of the 10%sample /FT. After 28 days, 0%/FT and 10%/FT showed compressive strength of 14.3 ± 0.5MPa and 13.3 ± 1.4 MPa, respectively, with no statistically significant difference. This can be attributed to the presence of fine glass particles, which, in addition to favoring mortar compaction, possibly had a good interaction with the FT/cement. Such results can contribute to the management of residual waste, reduction of cement and the reduction of the impact on the environment.

Acknowledgements:
The authors acknowledge PIBIC/UFPI.

References:

[1] M. Haque et al. Case Studies in Building Materials, 14, e00505 (2021).

pH effects on zeta potential and antibacterial activity of chitosan-based nanomicroparticles

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Chitosan (CS)-based materials have emerged as a promising alternative to replace toxic chemical compounds. These materials exert antibacterial activity (AbA) against phytopathogens [1]. The antibacterial properties of CS nano-microparticles (CS-NP/MP) depend on several intrinsic and extrinsic factors such as size and morphology of the CS- NP/MP, zeta potential (ZP), interacting pH, presence, and nature of functionalized compounds in the CS-NP/MP [2]. In this study, we describe the synthesis and physicochemical characterization of a set CS-NP/MP. Moreover, we evaluate the effect ofpH on the ZP of the CS-NP/MPs and its impact on their AbA. The particles were produced byionic gelation using tripolyphosphate (TPP) as a crosslinker, and during the synthesis process, different parameters were assayed (CS:TPP ratio, type, and agitation time). The obtained particles, designed as CS-NP, CS-MP1, and CS-MP2, exhibited similar sizes butcontrasting ZP values (13.9, 33.7, and 4.78 mV, respectively). Our results showed the ZP ofCS-NP/MP and its AbA against *Pseudomonas syringae*pv. *tomato*DC3000 decreases with increasing pH of the medium. Therefore, we describe an important role of pH to determinethe ZP of the materials and consequently the electrostatic interaction between the positivelycharged polymer and the negatively charged surface of the microorganism. We discuss ourfindings in light of the material properties and opportunities to use CS-NP/MP as a new typeof biomaterial in modern agriculture.

Acknowledgments

Supported by CONICET, UNMdP, and ANPyCT.

References

[1]. M. Chandrasekaran, K. D. Kim, and S. C. Chun. Processes, 8(9), 1173 (2020).

[2]. G. Geisberger, E. B. Gyenge, D. Hinger, A. Ka¨ch, C. Maake, and G. R. Patzke. Biomacromolecules, 14(4), 1010-1017 (2013).

Polysulfide from Inverse Vulcanization as a Multifunctional Fertilizer Material for the Controlled-Release of Phosphate

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In face of the concerning population growth predictions, food security is one of the biggest challenges of the next decades. Better plant nutrition is pivotal to increase food production and could be achieved sustainably with the development of smart environmentally-friendly fertilizers. Phosphorus (P) is vital for crop yields, but the use of P fertilizers is often associated with eutrophication of freshwater systems. To address this issue, fertilizer design plays a critical role. Sulfur(S) is also essential to plant metabolism, often managed with elemental sulfur (S8) application as a matrix to carry other fertilizers. Nevertheless, S8 matrices tend to be brittle and non-uniform, and the recovery of S8-S by crops depends on its biological oxidation to sulfate, which is significantly slow. Polysulfides (PolyS) are S-rich materials recently studied by our group as multifunctional S-fertilizers with superior S oxidation. In the present work PolyS was investigated as a matrix for the fine dispersion of P sources, forming controlled-release fertilizer (CRF) composites with enhanced phosphate and sulfate delivery PolyS is an ideal substitute to conventional CRF polymers, being an easily processible and malleable material with an agronomic role as a S fertilizer. The polymer was obtained with the inverse vulcanization of S8in the presence of an alkene (i.e.: soybean oil), a green one-pot method with no solvent use and great atom economy. The polymerization of S8crystalline structure produces an amorphous material with less ordered S-chains, proposed to favor the conversion to sulfate. Results showed that the matrix favored P dissolution while also preventing a fast release, therefore promoting an ongoing P-delivery. Moreover, soybean cultivation with the composites achieved higher S use efficiency, plant biomass, and root proliferation than with a commercial reference, proving PolyS potential as a versatile and efficient matrix for sustainable P and S fertilization. FAPESP 2018/10104-

Preparation of structured polymeric gels as a versatile nanocarrier for loading and controlled release of micronutrients for agriculture purposes

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The use of polymeric materials in agriculture and horticulture has seen a significant increase in recent years. Not only are they replacing traditional materials, but they are also providing a significant improvement in the technology used in the cultivation of crops and vegetables, the construction of storage for crops and animals, and in the equipment and drainage technologies used in agriculture.[1] Herein, a one-step preparation of polymeric gels was demonstrated by "click" characteristics of amine-epoxide reaction. The hydrogels, known as poly (amino alcohol ethers), was obtained via ring opening of epoxide group through the reaction of a polyetheramine (PPO) and poly (ethylene glycol) diglycidyl ether (PDE). The influence of molar ratio of PPO:PDE was evaluated. The final materials were characterized by DLS, SEM, thermal analysis (TGA). The cytotoxicity of the materials was evaluated by XTT assays. The hydrogels were biocompatible and the size of the particles can be fine tuning by the synthesis conditions. Seed germination (using corn seed as model) was evaluated using polymeric nanogels with Zn ions embedded. The preliminary result, demonstrate that structured gels could be used as novel technology for enhance seed germination and open an avenue of possibilities to be used as foliar applications for plant nutrition.

Acknowledgements: The authors are grateful for financial support provided by FAPESP (grants #2021/06552-1, #2022/06507-9, #2022/13408-7 and EMU #2021/14619-9), CAPES (Finance Code 001), and CNPq (grant #307696/2021-9).

References [1] F. Puoci, F. Iemma, UG. Spizzirri, G. Cirillo, M. Curcio, N. Picci, Am. J. Agr Biological Sci., 3, (1), 299-314 (2008).

Production of a hydrochar as a destination of saturated adsorbents

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The final disposal of natural adsorbents saturated with pollutants creates a major problem, as it is not always possible to treat or reuse such materials, resulting only in the transfer of pollutants from a fluid phase to a solid phase. [1]. In view of this, this work aimed to evaluate the production of biochar from kapok pods as a destination for this material, which was previously used as an adsorbent for the removal of rhodamine B (RhB) dye. The production of biochar was carried out by the hydrothermal method followed by thermal activation, using 5 g of the saturated kapok capsule. Two applications for hydrochar (HC) asa destination were evaluated: its use as an adsorbent also for RhB, and its direct disposal asa soil conditioner. Through the analysis of the pH of zero charge, it was observed that the neutrality region of the HC occurs at pH 6. Affinity and pH influence tests were carried out, using RhB at 10 ppm and 0.1 g of HC as adsorbent, verifying excellent adsorptive power of the produced material, reaching removal superior to 92% in the different applied pH ranges, such performance being superior to the natural material in the same conditions. Being a promising alternative for the treatment of concentrated natural adsorbents. Tests of application of the material as a soil conditioner were carried out using a variation of 4 to 10% of HC in the soil composition and it was observed that the material did not present any deleterious effect, since the seedlings presented satisfactory development in all samples, with growth greater than 60 cm in shoot length, 41 cm of radicle and 4 leaf units, in all cases studied. Both destinations for HC being excellent alternatives.

Acknowledgment:
CAPES/Brazil

References:

[1] Vasconcelos et al. Rev. Virtual de Química, no prelo, 1-28 (2023) (http://dx.doi.org/10.21577/1984-6835.20230017)

Production of chitosan hybrid films with açaí extractand evaluation of antibacterial activity

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The development of biodegradable materials is seen as an alternative in reducing environmental damage caused by the disposal of products based mainly on synthetic polymers. Aiming at this, chitosan is a polysaccharide with physicochemical characteristics and biocompatible with biological systems, having potential for the development of carriers of bioactive substances. With this in mind, the present work aimed to obtain and characterize hybrid polymeric films containing açaí (Euterpe oleracea) extracts. The films were produced by the casting method (mixture of chitosan, cationic mineral clay, polyethylene glycol and açaí), using the açai pulp aqueous extract and the acidified aqueous extract. The films obtained were characterized by SEM, FTIR, microbiological and antioxidant (DPPH) activity techniques. The SEM showed films with a surface of compact appearance, with cracks and small structures similar to holes. The FTIR analyses bring information of interactions between the polymer and clay, also revealing possible intercalations of phenolic compounds inside the hybrid film. The minimum inhibitory concentration (MIC) evaluation showed inhibition efficiency in both types of extract, highlighting a better activity in the acidified extract for the four strains evaluated. The microbiology through disc diffusion technique showed similar results in relation to the acidified extract. The results for the antioxidant capacity reveal that the acidified açaí extract presented capacity to inhibit the free radical, as well as the films, showing that afterthe polymerization process, to obtain the hybrid films, the biological properties of the extracts were maintained. The set of results presented reveals very effective and promisingfilms for future use of these systems in wound treatment.

Acknowledgments:
UFAL, IFAL, FAPEAL, CAPES, CNPq, FINEP.

Synthesis and characterization of PVA and EDTAD hydrogel for release of NPK nutrients in soil

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Abstract

According to FAO and CMA, in 2050 the world population will reach more than 9 billion people and to meet this growing and increasingly demanding demand for food, it will be necessary to increase food production by at least 60%. In this way, innovation will be crucial for sustainable food production, that is, without soil degradation and water waste [1-2]. The increase in agricultural production, without innovative solutions for replenishing nutrients in the soil and reducing water consumption, inevitably results in soil depletion and evaporation of up to 50% of the water intended for irrigation. To reduce soil depletion and at the same time increase agricultural production, preserving thousands of hectares of forests and native forests, as well as fauna and flora, a large amount of fertilizers are currently used. Thus, the objective of this work was to synthe polyvinyl alcohol (PVA) and ethylenediaminetetraacetic dianhydride (EDTAD) hydrogels, characterize them through FTIR, SEM and biodegradation analysis, in addition to evaluating their kinetics of absorption and release of water and NPK nutrients , through UV-VIS and flame photometry techniques. Through the analyzes carried out by FTIR, it was verified that the hydrogel was successfully synthed, observing the expressive increase of the free OH band at 1320 cm-1, the bands present at 1809 cm-1 and 1755 cm-1 referring to the anhydride of The carboxylic acid present in EDTAD is non-existent in hydrogels due to ring opening for intermolecular hydrogen bonding, which results in cross-linking of the chains. The kinetics of absorption and release of NPK was built, through which the feasibility of applying the hydrogel to release NPK and rational use of water was proven.

References

[1] http://www.fao.org/news/story/pt/item/283456/icode/. Acesso em 14/02/2019.

[2] http://dev2.worldwatercouncil.org/fileadmin/world_water_council/documents_old/Library/WWVision/Chapter2.pdf. Acesso em 14/02/2019.

Synthesis of Green Coating Polymer for Seed pre-inoculation: Improvement of Biological Nitrogen Fixation with *Bradyrhizobium spp*.

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Glycerol is being investigated as an alternative and environmentally friendly feedstock for polymer synthesis. As a trifunctional alcohol, its use in polycondensation reactions is particularly appealing [1]. Polyglycerol Citrate can be formed when citric acid and glycerol react (PGCit). Because both monomers are biodegradable and nontoxic, their management is dictated by ecology and green chemistry principles, and the synthesis can be done by polycondensation with water as the only by-product [2]. Furthermore, despite numerous studies demonstrating the ability of beneficial microorganisms to increase plant development, few bioinoculants adhered to seeds are commercially available because maintaining viable microorganisms during seed treatment and storage is a major problem [3]. To address this issue, the current study suggests using PGCit as a new type of inoculant vehicle for coating soybean seeds with ***Bradyrhizobium***. Furthermore, PGCit can be solubilized in liquid inoculant formulations, making seed application simple for farmers. The ^{1}H NMR results confirmed that it could be made in a simple one-pot polycondensation yielded in the PGCit structure, with some water solubility as desired. ***Bradyrhizobium***survival was increased by seed coating with PGCit on seeds stored for more than 14 days, resulting in successful bacterial and plant colonization. Greenhouse studies have confirmed their beneficial effect on soybean, while the polymer coating protects bacteria and seeds from abiotic stress in the environment and promotes successful inoculant colonization based on the number of nodules counted after harvesting the soy plants. As a result, the polymer proposed showed to be a promising eco-friendly approach to improving soybean production via the use of a microbial inoculant.

Acknowledgments

FAPESP grants 2016/10636-8, 2018/10104-1, and 2021/13645-6.

References

[1] 10.1021/acs.oprd.8b00306, [2] 10.1002/agj2.20392, [3] 10.1007/s00203-022-02779-z.

THERMAL DEGRADATION STUDY OF MICROSPHERES CONTAINING RYNCOPHOROL WITH SODIUM ALGINATE BY EXTRUSION METHOD

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Insect pests cause great economic losses to Brazilian agricultural plantations [1]. Thus, producers excessively use insecticides to avoid damage to the production chain[1]. R. palmarum is the most important pest of coconut cultivation, as studies on these insects have been carried out for decades on the invasion and destruction of plantations [1]. Currently, plastic sachets and Eppendorf polyethylene tubes are used as rhynchophorol releasers, potent aggregation pheromone[2]. The objective was to study the thermal degradation of microspheres containing rhynchophorol with sodium alginate by extrusion. The characterization of the microspheres was performed by thermal analyses, Thermogravimetry (TG), and Differential Scanning Calorimetry (DSC). According to the TG analyses, for treatment H, the mass loss was 19.152% from 26.02 to 215.90 ºC, in the first moment, and after, the mass loss of 52.486% from 215.90 to 531 .53°C. As for the DSC results, there was a slight exothermic event, around 132 °C, and a very sharp peak for an exothermic event around 485 °C. These data mean, respectively, the loss of volatile compounds and the decomposition of alginate and rhynchophorol. It is worth mentioning that the formation of microspheres of the rhynchophorol pheromone by the extrusion technique with sodium alginate and calcium chloride has not yet been reported in the literature, which demonstrates the originality of this work.

Thanks: The Federal University of Alagoas - UFAL and the Federal Institute of Alagoas - IFAL.

References: [1] CYSNE, A. Q. ; CRUZ1, B. A. ; CUNHA, R. N. V. e ROCHA, R. N. C. Flutuação populacional de Rhynchophorus palmarum (L.) (Coleoptera: Curculionidae) em palmeiras oleíferas no Amazonas. Acta Amazonica, v. 43, n. 2, p. 197-202, 2013. [2] BIOCONTROLE - Métodos de Controle de Pragas, 2017. Avaiable: https://biocontrole.com.br/. Acess at: 01 jan. 2023.

U-Sustainable Production of Nanomaterials and Ink Formulations for Affordable, Efficient, and Reusable Printed Electronics

Additive manufacturing of batteries: Comparison between fabrication processes

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At the moment, emerging applications and devices such as biomedical sensors or micro-electro-mechanical systems, are limited in shape, size and price by the energy storage devices that power them. In this regard, printing technologies propose an affordable and adaptable alternative of creating complex architectures that could maximize the energy density of batteries and supercapacitors[1]. Although a substantial effort has been done to push forward the application of printing strategies for energy storage devices, this field is still in its infancy and a lot of challenges remain[2]. A recurrent problem in this field is the lack of information on how the micro-structure of the composites is affected by the fabrication method and how this impacts the performance of the final device.

In this work, we present a systematic study of the changes in the electrode materials when we move from traditional techniques to printing-based methods. We use ink-based techniques encompassing traditional battery fabrication methods such as slurry casting and filtration and more advanced ones such as extrusion printing, spray coating and aerosol jet printing. The methodology used for the electrochemical analysis was mainly the analysis of the capacity-rate curves, that were fitted to physical models[3]. The differences between deposition techniques were correlated to the observations made by morphological and structural-based techniques, such as TEM, SEM and BET.

It was found that the deposition technique changes the conductive network arrangement, creating segregated[4] or randomly arranged networks. It was also found that porosity and out-of-plane conductivity are other factors that can be massively affected by the fabrication technique and its effect on the electrochemical performance is very significant.

[1] Y. Pang, et al Adv. Funct. Mater. 2020
[2] P. Chang, et al J. Mater. Chem. A 2019,
[3] R. Tian, et al Nat. Commun. 2019
[4] S.-H. H. Park, et al Nat. Energy 2019

Evaluation the environmental impact of electronics and how printed electronics can improve sustainability

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For more than 50 years, advances in electronics have revolutionized all socioeconomic sectors. However, in pursuit of this goal, the industry has evolved whereby the materials and manufacturing processes are decided by the technical and economic requirements, and not sustainability, resulting in poor practices and unintended outcomes. This presentation will first review some of the drivers and opportunities for sustainable PCB manufacturing, and then target specific areas of processing to induces changes that could improve the sustainability of electronic assemblies. Process modelling has been undertaken, and environmental assessment conducted, to support the final recommendation to improve the sustainability of the product. As such, a life-cycle assessment (LCA) was performed on a simple Printed Circuit Boards (PCB) made using the traditional manufacturing approach with FR-4, polyimide and paper-based substrates and this can be used to review the environmental impact at each key stage of the life cycle. The scope was cradle to grave, including embodied impacts, transportation, energy used during manufacturing, waste material generated and disposal. The impact of different processing steps was considered, all scored using the ReCiPe Endpoint. Results show that consideration should be given to PCB design, including number of layers used in multilayer boards. We also report on how solder and die attach material has a choice has an impact upon environmental profile. Generally, we find lower temperature solders and die attach materials lead to improve environmental impacts as less electricity is used, coupled with the small volumes of attach materials used. Finally, we show the move towards additive manufacturing and the use of alternative material and processes can reduce the environmental impact of electronic products which is validated through Life Cycle Assessment (LCA) calculations.

Sustainable Devices: From Ink Formulation to Their Printability

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The advent of portable electronics has undoubtedly brought convenience and value to our lives. However, the decreasing lifespan of these devices has led to increased pressure on e-waste management. Therefore, it has become imperative that future electronic devices should be designed based on sustainable principles to mitigate this issue[1–3]. The use of additive manufacturing techniques, particularly printing techniques, and the development of nanomaterials can lead to the creation of low-waste and eco-friendly products. Additionally, device implementation in eco-friendly substrates, such as paper and other cellulosic materials, further promotes the sustainability of modern industrial production methods.

In our research, we have provided a comprehensive analysis of the advancements made in the development of nanomaterial inks and their application in the printing of electronic components. Specifically, we have discussed the creation of ink formulations for metal oxides and low-dimensional materials that are both cost-effective and sustainable. Our focus has been on two main approaches: solution combustion synthesis (SCS) and liquid phase exfoliation (LPE)[4,5]. We have noticed a correlation between ink properties, such as viscosity, material concentration, and the physic-chemical properties of active materials, with their printing capabilities. Therefore, it was necessary to adjust the rheology of the inks to achieve functional printed devices. In this talk, we discuss the printing techniques and the printability of functional inks, along with the challenges associated with them. Finally, we have provided an outlook on the future of sustainable printed electronic applications.

[1] C. Larsen, P. Lundberg, S. Tang, J. Ràfols-Ribé, A. Sandström, E. Mattias Lindh, J. Wang, L. Edman, *Nat Commun* **2021**, *12*, DOI 10.1038/s41467-021-24761-x.

[2] W. Li, Q. Liu, Y. Zhang, C. Li, Z. He, W. C. H. Choy, P. J. Low, P. Sonar, A. K. K. Kyaw, *Advanced Materials* **2020**, *32*, 2001591.

[3] S. L. Silvestre, T. Pinheiro, A. C. Marques, J. Deuermeier, J. Coelho, R. Martins, L. Pereira, E. Fortunato, *Flexible and Printed Electronics* **2022**, *7*, 035021.

[4] E. Carlos, J. Deuermeier, R. Branquinho, C. Gaspar, R. Martins, A. Kiazadeh, E. Fortunato, *J Mater Chem C Mater* **2021**, *9*, 3911.

[5] S. Pinilla, J. Coelho, K. Li, J. Liu, V. Nicolosi, *Nat Rev Mater* **2022**, *7*, 717.

Bio-sourced Nanomaterials and Solvents for Electronics and Energy Applications

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A global effort is being made towards the sustainable development of electronics and energy applications. A careful choice of materials can significantly reduce the environmental impact of each main component of electronic/energy devices. Environmental and human health impact of e-waste derived from consumerism requires a mindset change from linear to circular thinking. Similarly, whilst clean energy has been a focus for several governmental initiatives, many of such more sustainable devices for energy harvesting/storage still require working with toxic or critical chemicals [1]. In this context, Merck KGaA brought several solutions for the workflow development of electronics and clean energy devices. Among such, we will cover ourbio-sourced customized solvents for electronics/energy applications, our bio-sourced nanomaterials, and our R&D100 awarded inks for more sustainable manufacturing [2]. We will describe how these more sustainable solutions do not compromise the reliable performance of your device. For instance, our bio-sourced high surface area (1155 ± 69 m2 /g) single laye sheets graphene for battery has an extremely high pore volume (1.7 ± 0.22 cm3 /g), enabling maximum energy power and density, as wellas fast charging and no degradation over thousands of cycles. The capacity of our anodeafter charging at 10 C (minutes) is similar to graphite capacity at a charge rate of 0.1 C (i.e.10 hours charging time). It can charge in a few minutes instead of hours and showed a 5-fold increase in capacity at 0.1C vs. graphite anode (standard).

Acknowledgements:We thank Swetha Chandrasekaran and Marcus A. Worsley from Lawrence Livermore National Laboratory, USA for the partnership with ink results.

References:[1] I. Chakrabartty, K. R. Hakeem, Y.K. Mohanta and R. S. Varma, Clean Technologies and Environmental Policy, v. 24, p. 3237-3252, (2022).[2] B. Yao et al. Advanced materials, v. 32, p. 1906652 (2020)

Electrodeposition of manganese oxide on paper-based laser-induced graphene for sustainable supercapacitor fabrication

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The production and accumulation of electronic waste have become a significant problem, as most electronic devices use non-degradable materials. Therefore, sustainable fabrication methods and biodegradable materials are required to mitigate this problem. Laser-induced graphene (LIG) is a sustainable way to produce high-quality porous graphene electrodes that do not require the use of toxic reagents. Furthermore, LIG can be produced from organic, biodegradable substrates such as cork, cotton, and paper. Due to its high conductivity, flexibility, and porosity, LIG has been successfully employed as electrodes for biosensing and energy storage, namely supercapacitors Recently, flexible supercapacitors have attracted attention for applications as efficient power source in wearable electronics. In this work, we present the sustainable fabrication of supercapacitors using paper-basedLIG electrodes with electrodeposited manganese oxide (MnO). We produced the LIG by irradiating with CO_2laser a piece of Whatman paper grade 1 treated with a fire retardant. We deposited the MnO on LIG in a three-electrode cell using manganese acetate as a precursor (0.1 M). We fabricated a symmetrical supercapacitor using Whatman paper grade4 soaked in PVA/LiCl as the separator. XPS analysis indicated three oxidation states for theMn: possibly MnO, MnO_2, and Mn_2O_3. From galvanostatic charge/discharge curves, we obtained a maximum areal capacitance of 86.9 mF cm^{-2}(at 0.1 mA cm^{-2}). Moreover, the device presented a remarkably high areal energy of 7.3 μWh cm^{-2}. In summary, we produced supercapacitors using sustainable techniques and materials that are competitive with devices produced by conventional pollutant methods.

Acknowledgements:
FAPESP, INEO, CAPES, POSMAT

Laser-Induced Graphene for flexible tracks and biodegradable printed electrolyte gated transistor

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The huge amount of electronic waste brings the need for sustainable electronic devices aiming to reduce environmental damage. This, coupled with the rise of the Internet of Things (IoT) creates a niche for a biodegradable printed electronic. For instance, it can be envisioned smart package with printed electronic circuits that allow monitoring of the product' s conditions exposure and states its quality. The main challenges in making this idea a commercial reality are the development of transistors and conductive tracks printed directly into the packages materials, as the paper. Laser-Induced Graphene (LIG) is an easy technique that stands up to produce graphene. Electrolyte-gated transistors (EGTs) offer high sensitivity, low voltage and power usage, making them perfect for flexible devices. LIG and EGTs show promise for flexible printed electronics and combining them is crucial for progress. Our study integrates LIG and EGTs, demonstrating their potential for flexible printed electronics. We use LIG to produce conductive tracks in kraft paper without any treatment and to produce transistor electrodes (gate, source, and drain). The tracks conductivity depends on LIG parameter as scan velocity and laser power. We achieved a conductivity of ~50 Ohms/sq, which is suitable for use as EGT electrodes and as track connectors. This work is shown EGTs on Kraft paper and in polyamide substrates using ZnO nanoparticles and PEDOT/PSS as active material. The electrode was produced in situ by LIG, the semiconductor was deposited by spray and the electrolyte was honey, dispensed by hand. We found the Ion/Ioff ratio of 103 and Ion on the order of 0.5 mA for the transistor in polyamide. Although in the paper the results are modest, they also show suitable for several applications. Also included pulsed characterization showing the neuromorphic application potential. This work shows the viability of producing printed EGTs on paper using LIG tracks and electrodes.

Piezoresistive ABS/CNTs Nanostructured 3D Printed Sensor for Industry 4.0

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The advent of Industry 4.0 based smart factories has resulted in a growing demand for intelligent sensors to enable the integration of cyber-physical systems across their production chains. This, in turn, facilitates the real-time automation and optimization of their processes [1]. Carbon nanotubes (CNTs) based nanocomposites are suitable for sensors, because of the processability of the matrixes and the properties of the CNTs [2]. Inthis sense, the present study developed a low-cost, nanostructured poly(acrylonitrile- butadiene-styrene) (ABS) smart sensor produced by additive manufacturing. After printing the samples by fused deposition modeling (FDM), a solution layer of acetone, dimethylformamide and CNTs functionalized with carboxylic acid was applied using an aerograph. Electrical measurements made with a model 6487 Keithley picoammeter after solvents' evaporation showed the electrical resistance drop from 1012 to 103 Ω after five layers of the solution. Furthermore, the samples behaved as a piezoresistive metamaterial, since their electrical resistance changed around 1 kΩ with physical deformations due to pressures of up to 0.27 MPa. Field Emission Gun Scanning Electron Microscopy (FEG-SEM) showed the nanostructured film spreading after each layer of CNTs as well as the interaction between the ABS substrate and the nanotubes. Raman spectroscopy also showed this interaction, as seen by the presence of characteristic bands of both on the nanocomposite Raman spectra. These results show the electromechanical response of the material, giving possibility to implement it with ESP32 microcontroller as smart sensor for IoT applications.

Acknowledgements:

Thanks to CAPES (nº 88887.600733/2021-00) and Finep (nº 01.22.0133.00) for financial support.

References:

[1] V. Alcácer and V. Cruz-Machado, Eng. Sci. and Tech. vol. 22, pp 899-919 (2019)

[2] V. Bezzon et al., Adv. in Mat. Sci. and Eng. vol. 2019, 21 pages (2019)

Production and characterization of graphene-based hybrid inks

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The field of materials science has seen significant advancements in the production and characterization of graphene-based hybrid inks. These inks are created by combining graphene with other materials, such as polymers or metals, to produce new materials with unique properties. The current paper proposes a method for producing hybrid graphene ink combined with copper and silver. The process involves synthesis, deposition, and characterization. Firstly, graphene oxide was synthed using the Hummers method. Then three main versions of the ink were prepared, including a control version without metals, a version with a low concentration of silver, and a version with a low concentration of copper. The material was then deposited onto microscope slides using the doctor blade coating method. The resulting samples were analyzed using various techniques, including light microscopy and mechanical profilometry to check for topography, homogeneity, thickness, and roughness. In addition, raman microscopy, X-ray diffraction, and infrared microscopy were used to verify crystalline quality and chemical structure. Otherwise, conductivity measurements were performed using the 4-probe and Arduino systems. Additionally, uv-visible light spectrophotometry, viscosimetry, and optical profilometry were used to investigate the inks optical properties. Scanning Electron Microscopy (SEM) and viscosimeter analyses were also performed for the analysis of additional properties such as viscosity and stability of the inks produced. The samples showed a homogeneous appearance, low defect density, thermal stability, and high conductivity, making them suitable for use in electronics, energy, and biomedicine.

Acknowlwdgements:

Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and Fundação deAmparo a Pesquisa do Estado de São Paulo (FAPESP)

A Study of a Hybrid Schottky Photodiode Based on ZnO/AuNPs and PEDOT: PSS to Enhance UVA Detection Through Plasmonic Effects

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ZnO is an n-type semiconductor with a direct bandgap of ~3.3 eV, making it an excellent candidate for application in UVA photodetectors. Furthermore, the insertion of gold nanoparticles (AuNPs) into the ZnO bulk can broaden its absorption spectrum by plasmon-induced charge separation (PICS). The Schottky diode (SD) based on the ZnO/PEDOT:PSS rectifying junction is an excellent alternative among several photodetector devices. Here, we investigated the effects of adding AuNPs into the ZnO bulk on the performance of a SD for application as a photodetector. Numerical simulations using the finite element method were employed to evaluate the SD's behavior based on ZnO/AuNPs in both dark and UV-illuminated conditions. For the experimental work, the devices were fabricated as follows: i) Al (~15 nm) was evaporated as the bottom electrode, followed by the deposition of ~80nm of ZnO via spray pyrolysi ii) incorporation of AuNPs-like structures by the deposition of Au (~1 nm) to form unconnected gold spot iii) spray deposition of additional ~20 nm of ZnO over the AuNP-like structure; iv) deposition of the PEDOT:PSS as the top electrode through spray coating. The morphological and optical characteristics of the ZnO semiconductor films, with and without the AuNPs within its bulk, were analyzed using UV-VIS, AFM, SEM-FEG, and others. The electrical characterization of the SD was carried out through DC and AC measurements. The I-V curve showed a rectification of 3 orders of magnitude in the dark, while the rectification practically disappeared under UV illumination. The AC measurement in C-V mode showed an improvement under UV light due to the presence of AuNPs. In summary, we confirmed enhancements of detection through the plasmon effect by incorporating AuNPs in the ZnO bulk. This study can potentially expand the scope of Schottky diode applications as a photodetector.

Acknowledgments
FAPESP (2021/01548-6, and 2022/12332-7), CAPES (001), POSMAT, and INEO

Development and Characterization of Biodegradable Sensors Based on ZnO

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The search for technological advances in recent years has attracted research into semiconductor materials, in which they have a fundamental role in electronics, due to their importance for the production of devices and sensors in the solid state. This work seeks to study the characterization and optimization of the preparation of thin film zinc oxide films obtained by the Sputtering Physical Vapor Deposition (PVD) technique, aiming to control its semiconductor behavior, as well as the fabrication of two-terminal devices by deposition of electrical contacts on the films. The characteristics of the electrical contacts in ZnO, whether ohmic or Schottky, depend not only on the conductors used, but also on the characteristics of the ZnO film itself, such as the doping density. In this study, the morphological characterization of the ZnO film will be carried out using X-ray diffraction analysis (DRX), scanning electron microscopy (SEM), X-ray fluorescence (FRX), UV-Vis, in addition to electrical characterizations of the two terminals as a diode depending on the electrical contacts. The electrical characterization will be studied using biodegradable electrodes such as molybdenum (Mo) and zinc (Zn) in order to develop a basic study for the development of biodegradable devices based on ZnO.

Acknowledgments:

References:

FU, Y.Q.; LUO, J.K.; DU, X.Y.; FLEWITT, A.J.; LI, Y.; MARKX, G.H.; WALTON, A.J.; MILNE, W.I. Recent developments on ZnO films for acoustic wave based bio-sensing and microfluidic applications: A review. Sens. Actuators B Chem. 143, 606-619, 2010

SHAIKH, S. K., GANBAVLE, V. V., INAMDAR, S. I. RAJPURE, K. Y. Multifunctional zinc oxide thin films for high-performance UV photodetectors and nitrogen dioxide gas sensors. RSC Adv. 6, 25641-25650 (2016).

Green synthesis of silver nanoparticles via mechanochemistry

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Nanotechnology has been widely disseminated, with nanoparticles (NPs) used for several applications, from medicine to energy. Metallic NPs have attracted great interest and have a wide range of applications, mainly to their unique features, such as characteristic absorption linked to the shape and composition of the nanoparticles. An example is silver nanoparticles (AgNPs) that present absorption around 400 and 500-700 nm for nanospheres and nanoprisms, respectively. They feature a small size, large surface area, low toxicity, low cost, and may provide a more environment-friendly nanomaterial.[1] Commonly, the synthesis of AgNPs uses materials that can generate toxic by-products, such as sodium borohydride, used as a reducing agent. The development of more sustainable synthesis is one aspect that emerges from the philosophy of Green Chemistry. This area provides discussions, criteria, and parameters to generate new theoretical and experimental knowledge in chemistry [2]. In this line of reasoning, one method that emerges to contribute to a greener synthesis is mechanochemistry. It presents a greener, more efficient, and innovative possibility to promote solvent-free syntheses through the supply of mechanical energy [3]. Thus, this work aims to synthesize AgNPs via mechanochemistry, free of solvents and toxic additives. Nanoparticles were prepared under grinding for 30 minutes at a frequency 25Hz and analyzed via UV-VIS spectroscopy and TEM. The results showed that AgNPs, with different stabilizers, were successfully obtained. Thus, mechanochemistry presents the potential to develop greener and unconventional methods to prepare nanomaterials.

Acknowledgements: UFPE, FACEPE, and CNPq.

References:
[1] FECHINE, P. B. A. (org.). Fortaleza: Imprensa Universitária, 2020.
[2] KEIJER, BAKKER and SLOOTWEG. Nature Chemistry. 2019, 11, 190.
[3] BALÁŢ, P. et al. Chemical Society Reviews, 2013, 42, 7571.

Influence of humidity on UV-assisted ZnO-basedSchottky photodiodes

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The printed electronic has shown excellent potential for developing sensors that can be incorporated into personal adornment, cards and package. Zinc oxide (ZnO) films, obtained from organic precursor solution or by printing nanoparticle dispersion, have been widely studied for their ability to sense ultraviolet radiation (UV), gases and vapors. The interaction of ZnO films with the molecules present in the air causes them to modify their electrical conductivity. This phenomenon is mainly caused by the adsorption and desorption of oxygen and water species on their surface. When UVA is incident on ZnO films in an atmosphere with controlled humidity, it is possible to investigate how humidity modifies the transport mechanism in a device. In this work, we present the fabrication and characterization of photodiodes based on a sprayed ZnO active layer in which the persistent conductivity effect related to humidity is studied. All measurements were conducted in an environment with controlled relative humidity (RH). The photodiode architecture used bottom aluminum electrodes and PEDOT:PSS as the top electrode, sandwiching the ZnO layer. After UV incidence (355 nm), the device underwent Current x Voltage measurements at different humidities. The obtained curves were used to extract the differential slopes (m), revealing an ohmic conduction behavior (m=1) at low RH, and the same device transitioned to a space-charge limited current (SCLC) behavior as the RH increased. These findings demonstrate that exposure to moisture induces changes in the conduction mechanism of ZnO, making it a promising candidate for gas sensing applications given the linearity of the parameters observed.

Acknowledgements:
CAPES (001), INEO, FAPESP (Process 19/03934-0), CNPQ, POSMAT

Metal-Insulator-Semiconductor (MIS) Structure Basedon Printed and Biodegradable ZnO/Electrolyte Junctionfor Capacitive Ultraviolet Photodetector

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Printing techniques offer a cost-effective way to fabricate electronic devices. Despite the challenges such as limited lifetime, poor resolution and inferior performance, printed electronics have gained prominence in niches such as printed sensors. ZnO is a versatile semiconductor for printed UV sensors due to its printability by various techniques such as inkjet and spray-coating. ZnO also presents biodegradability and low toxicity. Here, we propose a spray deposited ZnO junction with a biodegradable cellulose-based electrolyte ina metal-insulator-semiconductor (MIS) structured device using ITO electrodes, aiming capacitive photodetectors and phototransistor applications. By using impedance spectroscopy, we investigated how UV exposure changes the device's interfacial properties, evaluating the information on interface trap states, flat-band conditions and carrier concentrations. The capacitance-frequency curve displayed a plateau at low frequencies under zero bias due to the formation of electric double layer at the ZnO/electrolyte interface. With the voltage increase, the device was driven to the accumulation regime, increasing the capacitance to 0.3 µF at V=1V. Also, the dielectric loss starts to show two dielectric relaxation peaks, and a changed plateau shape. The extra peak can be attributed to artifacts such as edge leakage due to geometric factors and parasitic capacitances. A carrier concentration of $2.5\times10^{19}\text{cm}^{-3}$was estimated from a capacitance-voltage curve in the dark, which was increased to $7.3\times10^{21}\text{cm}^{-3}$during a 355nm UV exposure. C_{UV}/C_{dark}-voltage plot presented a maximum of ~50 at V≈0.2V. Finally, the MIS structure was applied as a phototransistor, and its dark and under-UV transfer characteristics presented a correlation with the MIS capacitor analysis. Our findings indicate that the proposed MIS-structured devices can be a promising approach for UV sensing applications.

Acknowledgements:
FAPESP (2020/12282-4, 2022/12332-7), CAPES, CNPQ, POSMAT, INEO

PLA@CNTs-Based Thermoresistive 3D Sensor

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Temperature sensors make up nearly 80% of the global sensor market and offer great potential for improving both conventional materials and developing new materials such as nanocomposites based on carbon nanotubes (CNTs), as these materials possess intrinsic physical properties that enable the portability and rapid response of temperature sensing devices [1]. In this sense, this study describes a based on CNTs sensor and created through additive manufacturing by the process of Fused Deposition Modeling. For this, two serpentine-shaped samples (dimensions: 20 × 30 mm) were 3D printed using poly-lactic acid (PLA), and one of them was coated with a paint made from multiwalled CNTs functionalized with carboxylic acid, acetone, and dimethylformamide using an airbrush. After applying eight layers of paint, scanning electron microscopy micrographs revealed a randomly dispersed CNTs layer over the PLA polymeric matrix. This interaction was also confirmedvia Raman spectroscopy, as the PLA@CNTs sample showed characteristic bands from bothmaterials. Two-point measurements were made using a model 6487 Keithley picoammetershowed an electrical resistance drop from 145 GΩ (only with PLA sensor) to approximately730 kΩ (PLA@CNTs sensor). Additionally, a thermoresistance test via two-point measurement using heating/cooling cycles demonstrated a reduction/increase in the electrical resistance of the PLA@CNTs sensor and its response (based on the variation of the relative change in electrical resistance) was −4.75 ± 0.66 % when varying the temperature from 35 to 45 °C, characterizing it as a thermoresistive sensor. Therefore, this device can be suitable for applications in IoT (Internet of Things) for monitoring of human body temperature in real time.

Acknowledgements:
Thanks to FINEP (MAUFA Agreement nº 01.22.0133.00) and MIDR (TED nº 10/2020) for the financial support.

References:
[1] B. F. MONEA et al., Sensors, vol. 19, n. 11, 2464 (2019)

Potential Use of Electrolyte-Gated Transistors for Molasses Detection in Honey

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Honey is a natural product produced by bees and has had an increasing impact on the economy. In general, Brazil has one of the largest production capacities of organic honey in the world, whose differential is the low contamination by pesticides and antibiotic residues, since the production is predominantly from native vegetation. However, this product is not free from food frauds that target illegal and often toxic adulterations of the product in search of increases in marketing profits, with an estimated 13% of honey samples in Brazil added with sugar syrup. Thus, it is of great interest to develop a sensor, cheap, easy to handle and interpret results, capable of detecting possible adulterations in honey in the apiary or meliponary itself through simple electrical measurements. In this work, progress towards the development of such a sensor based on electrolyte-gated transistors (EGTs) is reported, where a pure honey sample or a pure molasses sample is used as the liquid dielectric layer, placed between the gate electrode and the semiconductor channel. Transistors operations are validated and typical parameters, such as threshold voltage, on/off ratio, among others, are obtained. Two types of semiconductors are being investigated as channel, seeking for the best operating device's performance: (i) n-type zinc oxide (ZnO) thin films and (ii) p-type conjugated polymer poly(3-hexylthiophene-2,5-diyl) (P3HT), which increases the prospects of development of such a sensor.

Acknowledgements:

CAPES - Finance Code 001, MCTI CNPq A.B.E.L.H.A. - Processo 401522/2022-9, PIPE, FAPESP

References:

[1] D. H. Vieira, et. al. Applied Physics A, 2023, 129, 272

Production of Electrodes on Untreated Kraft Paper for Flexible Heaters via Laser-Induced Graphene Technology

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The production of flexible heating devices is an area undergoing constant evolution, with applications ranging from the textile industry to medicine. However, producing flexible electrodes with good heating properties and mechanical resistance presents a scientific and technological challenge. Graphene, a highly conductive and mechanically resistant material, has emerged as a promising choice for such applications. Laser-induced graphene (LIG), an easy form of graphene production using a laser to carbonize a hydrocarbon layer such as polyimide, has gained popularity. LIG can be applied to various substrates, including paper, which is cheap and biodegradable. However, most conventional techniques for producing LIG on paper require toxic and/or expensive chemical treatments, conflicting with its primary advantage. The use of untreated paper as a substrate is an affordable and safe alternative to produce graphene electrodes. In this study, we present the production of flexible electrodes using untreated paper as a substrate and their application in flexible heaters. Grid-shaped LIG electrodes were embedded in Kraft paper to manufacture heating devices. These devices can heat up to 30°C above ambient temperature in approximately 180 seconds under an applied voltage of 10V. We provide a comprehensive characterization of power and efficiency, considering the design layout and parameters used in the formation of LIG electrodes. The results show that flexible graphene electrodes using kraft paper as a substrate can be produced with excellent performance. Additionally, the produced heaters exhibit good mechanical resistance, making them a viable option for various sector applications. In conclusion, we present a new approach to producing resistant flexible heaters using graphene electrodes produced via LIG and untreated kraft paper as a substrate. This approach presents promising results and opens new possibilities to produce flexible heating devices safely and economically.

Thin Flexible Films Obtained by Slot Die Coating ofWater Based P3HT and P3HT:PCBM Nanoparticles

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With the slot die deposition technique it is possible to make much larger active areas than the spin coating technique with different substrates, allowing printing conductive inks in a similar way to a roll-to-roll printer inside laboratories and industries. Another feature that interferes with nanostructured devices scalability is the organic solvents for polymer inks that are highly toxic and harmful to the environment. In this way, the main goal of this study was the production of donor and acceptor nanoparticles in a stable suspension in water, which is a non-toxic and not harmful to the environment solvent, synthed by miniemulsion technique, obtaining an adequate dispersion for film depositions. We produced films for optical, chemical, morphological and electric characterizations, by slot die coating for a greater study of its properties. Besides that, slot-die films both in glass and acetate substrates were made for comparing the possibilities of transferring the technique to other types of flexible substrates. Measures for comparing the lifetime of the nanostructured and simple polymer based inks were also made. With the optical characterization it was possible to see the quenching between the donor and the blended nanoparticles of P3HT:PCBM, showing that there was a charge transfer between the donor and acceptor materials inside the nanostructure. The Raman spectrum of the P3HT and P3HT:PCBM nanoparticles showed that the chemical structure of the materials was not affected during the productionof the nanoparticles. The morphological study showed that the slot die coating printing methodology successfully produced an homogeneous film of the smaller nanoparticles withdiameters of approximately 30 nm.

V-Advanced nanocomposites for photonics : materials synthesis, optical properties and applications

Active aerosols to control the propagation of light

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This work connects the fields of active plasmonics and aerosols to establish an active aerosol. We report the experimental dynamics and control of ensembles of gold nanorods suspended in air by varying the magnitude and frequency of externally applied electric fields, thereby demonstrating an active aerosol. We further show light filter, valve and gradient-index responses, demonstrating active aerosols as a novel type of optical element we define as component-less optics.

Characterization of Novel Nonlinear Optical Materials

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Many materials, especially composites can exhibit nonlinear refraction (NLR) and absorption (NLA) that exhibit complex temporal dynamics. As a result, measurements made for one laser pulse width may not accurately predict behavior with different pulse widths. One solution is to make measurements with multiple pulse widths, but a better solution is to use excite-probe techniques. Our group has developed a method, Nonlinear Beam Deflection, (BD) that allows sensitive time-resolved measurement of both nonlinear refraction (NLR) and absorption (NLA). The measurement uses an excitation beam to create an index gradient in the nonlinear sample, which deflects a probe beam, ant the deflection is measured using a quad-cell detector in the far field. It has been shown that the method hasa sensitivity to induced phase changes as small as 1/24,000 of a wavelength. By changingthe relative polarization of the excitation and probe, we can also separate the bound- electronic responses from slower and different-symmetry nuclear contributions. In gases and liquids where reorientational nonlinearities are important, and measurements at the magic polarization angle allow isolation of the ultrafast nonlinearities. This method also allows for measurements of nonlinearities using very different wavelengths for the excitation and probe. We have used this method to characterize the impulse response function for third-order nonlinearities in many materials and has been the primary methodused to measure the properties of transparent conducting oxides. This allows accurateprediction of the nonlinear refraction and any pulse width longer than that used for the BDcharacterization. The method proves to be very useful in organic materials and nanocomposites that may show strong nonlinear absorption, as compared to other methods, such as Z-scan. We will show some new results that demonstrate how to maximize sensitivity via geometric optimization.

Earth-Abundant Layered Materials: VibrationalProperties and Nanophotonics Applications

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Phyllosilicates emerge as a promising class of large bandgap lamellar insulators. Their applications have been explored from the fabrication of graphene-based devices to two-dimensional (2D) heterostructures based on transition metal dichalcogenides with enhanced optical properties, optoelectronics, and polaritonics 1-4. Despite the extensive knowledge of the chemical composition of bulk phyllosilicates, retrieved from standard techniques such as Raman and IR spectroscopies, very little is known about those optical properties at the sub-micron spatial scale. Here, we provide an overview of IR s-SNOM for studying nano-optics and local chemistry of various 2D natural phyllosilicates including talc, phlogopite, and clinochlore5,6. Also, we bring a brief update on applications that combine natural minerals, graphene and phyllosilicate layers into functional heterostructures2,3. Additionally, we show that natural 2D talc supports hyperbolic phonon-polaritons (HPhP) similarly to the well-established 2D polaritonic hBN and α-MoO3 crystals in the far- and mid-IR range. Further, we highlight a few advances regarding nano-optical modulation in graphene-phyllosilicates devices driven by electrical control.

Acknowledgments:Fapesp (2019/14017-9), CNPq, Brazilian Nanocarbon Institute of Science and Technology (INCT/Nanocarbono) and CNPEM.

References:

1. Prando, G. A. et al. Phys Rev Appl 16, 64055 (2021).
2. Cadore, A. R. et al. 2D Mater 9, (2022).
3. Barcelos, I. D. et al. ACS Photonics 5, acsphotonics.7b01017 (2018).
4. Nutting, D. et al. Nanoscale 13, 15853-15858 (2021).
5. de Oliveira, R. et al. Appl Surf Sci 599, 153959 (2022).
6. Longuinhos, R. et al. The Journal of Physical Chemistry C, 127, 5876–5885(2023).

Fabrication and assessment of mechanically flexible 1D photonic crystals and active planar waveguides

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Thin-film optics is a key technology for the fabrication of miniaturized photonic devices, spanning from optical waveguides and photonic-integrated-circuits for optical signal processing, to multi-layered resonant structures and cavities for the confinement and spectral selection of the optical field. Active optical waveguides and photonic crystals are among the most versatile examples. One further step to add versatility to thin-film photonic structures involves the use of flexible materials. In fact, by adding mechanical flexibility to the rigid photonic systems, the range of applications greatly expands. However, passing from rigid to flexible substrates requires the development of suitable fabrication protocols, to preserve the optical and spectroscopic properties of the systems under mechanical deformation. We present the RF-sputtering fabrication of 1D photonic crystals and active Er^{3+} planar waveguides deposited on polymers and ultrathin flexible glass substrates. The structures deposited on ultrathin flexible glass show interesting results in terms of both optical and mechanical properties, making RF-sputtering a promising and scalable technique to fabricate flexible photonic devices [1,2]. Moreover, we report on the spectroscopic study of a 1D photonic crystal, fabricated via RF-sputtering on a flexible thermosetting polymer, in different bending conditions.

Acknowledgements:

This research is supported by the projects: FESR-PON 2014-2020 BEST4U ARS01_00519; CNR-PAS "Flexible Photonics" (2020-2022); NAWA PPN/IWA/2018/1/00104; MIUR- 'Departments of Excellence' L 232/2016; ERC-H2020 PAIDEIA GA 816313; "nuovi Concetti, mAteriali e tecnologie per l'iNtegrazione del fotoVoltAico negli edifici in uno scenario di generazione diffuSa" CANVAS and NAWA-MAECI Canaletto (2022-2023).

References:

[1] A.Carlotto, et.al., Proc. SPIE 12142, pp. 1214206 (2022); doi:10.1117/12.2621281

[2] A.Carlotto, et.al., Ceramics International, (2023); doi:10.1016/j.ceramint.2023.03.012

Functional nanostructured tungsten oxides films by RF-sputtering

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Polymorph tungsten oxide and its sub-stoichiometric phases (WO_{3-x}) have an extraordinarily broad range of possible photonic applications, from electrochromic layers in smart windows, to UV optical detectors, full-optical chemical sensors and photo-electro-catalytic membranes. We focus on Radio-Frequency (RF) magnetron sputtering deposition as a broadly versatile method to synthesize WO_{3-x} in different stoichiometries and nanostructures, easily up-scalable to industrial processes. We report on WO_{3-x} films fabricated under different recipes, which deeply differ in terms of possible applications. Optical-grade compact WO_{3-x} films can be fabricated by non-reactive magnetron RF-sputtering deposition from a WO_3 target at room temperature, followed by thermal annealing to tailor stoichiometry and structure. Films transparent in the NIR and electrically conductive can be obtained, that perform well as transparent contacts [1], but also insulating films, with the possibility to embed them into multi-layered IR resonant photonic structures for opto-chemical sensing [2]. Nanoporous WO_{3-x} photoelectrodes for green H_2 production are obtained from metallic W target, by RF-plasma sputtering process in a diode configuration in a reactive 40% O_2/Ar atmosphere. By changing the total gas pressure in the growth chamber, at same partial O_2 pressure, a bilayered diode-like structure is formed, which improves photoelectron transfer and decreases the interfacial resistance (Rct), leading up to about a 30% increase in photo-electro-catalytic (PEC) performance compared to monolayers coatings and to a 93% faradaic efficiency, which is among the highest reported so far for WO3 photoanodes [3].

[1]H. Chen et al., Proceedings SPIE 12142, 121421J, (2022).
[2]H. Chen, et al., Optical Materials X, Vol. 12, (2021).
[3]G. L. Chiarello et al., J. Mater Chem A, 5, 12977, (2017).

Acknowledgements: European fund - FESR, PON Ricerca e Innovazione 2014-2020, Project BEST4U -ARS01_00519.

Incorporating specific nanoparticles to induce new functionalities in optical fibers

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Engineering silica optical fibers by nanoparticle doping is a promising technology that allows the introduction of new functionalities and extends their applicable fields. However, the knowledge gap about the impact of the extreme fabrication temperatures on the nanoparticle features prevents the field progress. Herein, we demonstrate that the particularities of fiber fabrication, such as fast-heating rates and quenching heat treatments, can be leveraged to explore unlikely phenomena at the nanoscale under standard laboratory conditions. We have explored the incorporation of several spherical nanoparticles such as Ca-, Sr-, Ba-based nanoparticles and more recently cubic-shaped and rod-shaped YPO4 nanocrystals. Structural and anisotropic differences allow engineering differently their shape and composition in the fiber core by tailoring the drawing temperature, as revealed by a thorough study consisting of scanning electron microscopy (SEM), high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM), electron energy loss spectroscopy (EELS), and high-resolution transmission electron microscopy (HRTEM). During this presentation we will show the possibility of doping optical fibers fabricated by modified chemical vapor deposition (MCVD) and the stability of the specific formed phases. These findings open up new avenues to study shape-dependent properties of rare-earth orthophosphate (REPO4) nanostructures, among others, in optical fibers which will allow incorporating unprecedented functionalities and will have an impact in several fields of application, such as fiber lasers and optical fiber amplifiers, among others.

Managing Nanomaterials for Boosted Random Lasers Performance: Basics and Multidisciplinary Applications

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Random Lasers (RLs) are low coherence optical sources whose optical feedback mechanism responsible to sustain laser oscillation arises from light scattering in disordered nanostructures, instead of two set of fixed mirrors as in conventional lasers. Therefore, RLs rely heavily on managing nanomaterials for optimum performance. In this talk, I shall review the basics of RLs and the role of nanomaterials in their design. Recent developmentsand multidisciplinary applications will be highlighted.

Nanomaterials properties for photonics applications

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Nanomaterials have attracted a great deal of attention due to their increasing number of photonic applications. Here, we investigate the optical properties of various nanomaterials for applications in random lasers, nonlinear devices, and plasmonic biosensing. The materials studied are quartzite, as scatters in random laser (RL) [1], silica-based systems for plasmonic biosensing, and an organic crystal for second harmonic generation (SHG).

In the RL configuration, with a concentration of 0.5mg/mL Quartzite/Rh6G in Ethyleneglycol, we verified a decrease of 91% in the FWHM, with a threshold energy of ~0.3mJ. We also performed simulations to explore the geometry of nanoparticles for RL systems.

For potential applications on biosensing, we studied the optical properties of systems composed of gold nanoislands biotin-functionalized to detect avidin, at the surface of silica (SiO2) xerogels monoliths.

An average redshift of 52 nm was observed in the Localized Surface Plasmon absorbance peak of all tested concentrations.

For the nonlinear optical characterization of a nanomaterial, a crystal built up from 2-aminopyrazine as a cation, with N1 protonation and dihydrogen phosphate as anion [2] was probed with Nd:YAG laser (1064 nm, 900 mW). The SHG light was detected and the FWHM of the 532nm pulse train was ~1.33 shorter than that of the IR pulses. Also, an angular dependence of the SHG light intensity was observed.

Acknowledgments:

I.C.S.C. acknowledges financial support by, CNPq, CAPES, and National Institute Photonics (INCT de Fotônica) and the support by the ONRG.

References:

[1] Anderson S.L. Gomes, André L. Moura, Cid B. de Araújo, Ernesto P. Raposo, Progress in Quantum Electronics 78, 100343 (2021).

[2] A. K. S. M. Valdo, A. M. Sarotti, F. T. Martins, Cryst.Eng. Comm 19, 5960(2017).

Photonic Nanostructures for Energy, Sensing, and Photothermal Applications

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This presentation will describe the fabrication and characterization of a variety of photonic nanostructures and their emerging applications. Examples will include (1) Au nanoparticles, Ag nanoparticles, and hollow Au-Ag nanoshells as supercapacitor materials, (2) Au nanostars, Ag nanostars, and hollow Au-Ag nanostars for sensing, and (3) various photonic nanostructures with near-infrared extinctions for photothermal modulation of shape-memory polymeric structures. Details of the synthesis of these materials will be discussed along with details of their structural characterization, optical characterization, and photonic-driven materials performance. The exploration and development of these photonic systems will not only expand our fundamental understanding of the relationships between nanoparticle structure/composition and photonic response, but will also lead to advances in nanoparticle-based applications in energy, sensing, and photothermal modulation.

Photonics at high-temperature regimes with refractory materials

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Refractory metals and dielectrics are a promising material alternative for optical devices that operate at ultra-high temperatures because of their high-temperature stability and suitable optical properties. Yet, most prior reports have used their room-temperature permittivity when designing structures and characterizing their performance. We present a comprehensive analysis of the optical properties of refractory materials for high-temperature photonic applications, including three examples. First, we experimentally demonstrate vivid color pixels with potential stability >1,000 oC [1]. Second, we introduce a paradigm for materials' screening for thermophotovoltaic optical emitters, where we identify combinations that enable theoretical power conversion efficiency >60% [2]. Third, by combining in situ experiments up to 1,500 oC with calculations we present some examples of optical emitters and superabsorbers that combine high thermochemical stability with excellent optical behavior [3]. Overall, the use of refractory materials is enabling photonic devices that are stable under extreme temperature conditions.

References:

1. Duncan, M. A., Barney, L., Dias, M. R. S. and Leite, M. S. "Refractory metals and oxides for high-temperature structural color filters," ACS Appl. Mater. Interfaces, Vol. 14, 55745–55752, 2022.
2. Dias, M. R. S., et al. "Photonics roadmap for ultra-high-temperature thermophotovoltaics". In review, 2023.
3. Gong, T., Duncan, M. A., Karahadian, M., Leite, M. S., Munday, J. N. "Broadband superabsorber operating at 1,500°C using dielectric bilayers". In review, 2023.

Recent advances in engineering nanoparticles inoptical fibers

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Among the diversity of existing materials studied, nanoparticles-containing optical fibers are studied for the realization of lasers and sensors [1]. All these applications depend on our ability to control the characteristics of the nanoparticles in the fibers. To achieve this goal, we will discuss on two routes, both based on preforms prepared by the MCVD process. First, we will discuss on a route to obtain crystalline particles in as-drawn fibers [2]. Here, solution doping was used to embed YbPO4 crystals in the silica soot. We demonstrate, using scanning/transmission electron microscopes and confocal Raman microscope characterizations, that YbPO4 crystals not only survive the MCVD process but also the drawing process despite the high temperature involved (up to 2100 ◦ C) during fabrication processes. The second route deals with the control of the drawing parameters. It allows to control the shape and the size of the nanoparticles as they can elongate or break-up depending on the drawing temperature [3]. All these results allow to achieve a real engineering of the characteristics of the nanoparticles.

References
[1] W. Blanc et al., Progress in Materials Science 134 (2023) 101084
[2] Z. Lu et al., Optical Materials 138 (2023) 113644
[3] M. Vermillac et al., Journal of the American Ceramic Society 100 (2017) 1814-1819

Relationship between structure and nonlinear optical response in nanostructured materials

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Nanostructured materials have attracted considerable attention for their optical properties, in particular for their large nonlinear response, and their potential for different applications such as information processing, and biological and chemical sensing, to name a few. The main reason for this is the ability to tune the optical properties by manipulation of the composition, structure and symmetry of the composites. For second-order, and to a lesser extent also for third-order nonlinear processes, the response is highly dependent on the symmetry of the material structure. It is this dependence of the response on material symmetry that makes nonlinear optics itself an interesting tool for probing the nanoscopic structure of the materials by means of merely macroscopic measurements. In this work we present results for the study of the structure of different materials employing nonlinear measurements. On one hand we present a study of second harmonic generation in elongated metallic nanoparticles embedded in glass, which have random positions but are aligned in a preferential direction. On the other hand, we also present second harmonic generation and nonlinear absorption studies in hexagonal arrays of metallic nanoprisms. In all cases the polarization dependence of the response observed is related to the structure and symmetry of the materials, through modelling of the nonlinear response, finding that we can indeed relate the observed response to the nanoscopic structure of the materials studied.

Second Harmonic Scattering of Nanoscale Materials : From Incoherent to Coherent Scattering

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Second Harmonic Generation (SHG) is encountered in its standard form in macroscopic non-centrosymmetric materials where phase matching drives the resulting intensity. At the nanoscale, phase matching no longer dominates and one can access fine details of the material quadratic susceptibility. However, the breaking of the phase matching condition enables as well the possibility to observe SHG scattering. The loss of coherence nevertheless is not complete. The presentation will address this question. In a first part, I will discuss incoherent SHG scattering with nanoparticles dispersed in a liquid solvent. In the particular case of nanoparticles, I will explore further the delicate balance between composition, size and shape and propose potential applications [1, 2]. I will further question the possibility of single nanoparticle measurements with control over the nanoparticles, opening new possibilities for applications. In a second part, I will address the case of the existence of a coherent contribution besides the incoherent one. To this purpose, I will describe the case of neat liquids and show how short range interactions can be accessed using SHG scattering [3].

[1] L. Bonacina, P.F. Brevet, M. Finazzi, M. Celebrano, Harmonic Generation at the Nanoscale, J. Appl. Phys., 127 (2020) 230901.
[2] J. Duboisset, P.F. Brevet, Second Harmonic Scattering Defined Topological Classes for Nano-Objects, J. Phys. Chem. C, 123 (2019) 25303.
[3] J. Duboisset, P.F. Brevet, Salt induced Long-to-Short Range Orientational Transition in Water, Phys. Rev. Lett., 120 (2018) 263001.

A smart nanoplatform based on upconversion nanoparticles functionalized with europium complexes for temperature sensing

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Europium β-diketones (Eu^{3+}) complexes are known to exhibit interesting luminescent properties in the red region, making them excellent candidates for bioimaging or temperature sensing purposes [1]. However, their practical use is, sometimes, compromised due to their solubility and their prominent excitation by ultraviolet radiation [2]. Thus, the objective was to develop a smart nanomaterial based on the anchoring of the [Eu(TTA)3ephen] complex on the surface of upconversion nanoparticles (UCNP) doped with thulium ions ($LiYbF_4:Tm_{0.5}@LiYF_4$) and to evaluate the energy transfer processes between the particle-europium complex. For this, UCNP was obtained by the thermal decomposition method, followed by a coating with a thin layer of silica, which was later functionalized with the luminescent complexes. Overall, UCNP showed high-homogeneity with an average diameter of 30 nm after silica coating. XRD, TEM, EDS, FTIR, XPS, and zeta potential confirmed the structure and functionalization of the nanoparticles. By photoluminescence, UCNP showed characteristic emission of the Tm^{3+} ion in the ultraviolet, blue, red, and near-infrared regions when excited at 980 nm. In the presence of the Eu^{3+}-complex, UV-thulium emission disappearance since these photons are absorbed by the ligands coordinated to the Eu^{3+} ions that act as an antenna promoting their subsequent red emission. Initial tests between 15 and 50°C indicated that the sample has potential use as a two-center ratiometric thermometer since the luminescence intensity ratio (LIR) between different Tm^{3+} and Eu^{3+} transitions resulted in high relative thermal sensitivities of up to 1% $°C^{-1}$ at 50 °C.

Akcnolegdments

CAPES (88887.695875/2022-00) and FAPESP (19/26103-7).

References:

[1] George, T.M.; Krishna, M.S.; Reddy, M.L.P. Dalton Trans., 45, 18719, (2016).

[2] Reddy, M. L. P.; Divya, V.; Pavithran, Rani. Dalton Trans., 42, 15249, (2013).

Automated machine learning to improve the accuracyof luminescent thermometers

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Luminescent materials have been investigated for optical thermometry under different underlying physical mechanisms, and most of the researchers have been focusing on the seeking of new functional materials in order to improve the sensitivity and thermal resolution of thermometers to mainly spread its application for a clinical level. However, the accuracy of those thermometers is still considered unsatisfactory for applications at the nanoscale. As we show in the manuscript, the uncertainty, especially in real-time measurements, can be due to intrinsic fluctuations of the spectrometer from one acquired spectrum to the next. Those fluctuations are present even for large signal-to-noise ratio. Then, we exploited Automated ML (AutoML) tools to search in a wide range of models and hyperparameters and identify the optimal solution for luminescence thermometry via genetic programming. The best ML algorithm provides optimal responses (larger than 5.5×) when compared with traditional luminescence thermometry methods. As a proof-of-concept, we investigated low sensitivity Nd3+ doped YAG particles under excitation at 760 nm and the results pointed out that using artificial intelligence is the direction for a possible clinical-level application of luminescence nanothermometers. Although tested here only for low sensitivity Nd3+ doped YAG particles, the methodology can be applied to any photoluminescent material and excitation wavelength. Therefore, in order to make our findings accessible to a broad audience, we made our algorithm open-source code, which was designed to be accessible to individuals who have no programming experience, with the goal of facilitating reproducibility and extension.

Co/Nb modified barium titanate synthesis via sol-gel method for transparent photonics applications

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Barium titanate is a lead-free perovskite that can present numerous interesting properties for a variety of applications, from bioengineering to photonics. Regarding the last one, barium titanate is known for yielding photoelectric effect [1], although its wide band gap restricts its efficiency. This wide band gap actually represents an obstacle for efficient photonics applications, thus it is imperative to narrow its band gap to achieve more prominent results. The present work aimed to modify barium titanate band gap by introducing both cobalt and niobium precursors as an attempt to dope its lattice and study band structure modification. The synthesis was made via sol-gel, and the samples were analyzed by XRD, SEM and HR-TEM imaging, UV-Vis, FTIR and Raman spectroscopies. XRD data showed a structure modification from tetragonal in the pure sample to pseudo-cubic in modified samples due to oxygen vacancies stabilization [2], also presenting peak shifting that indicates lattice doping. From UV-Vis spectra it was possible to notice that classic indirect band gap was not changed for all samples, remaining at 3.5 eV, although direct band gaps with values between 1.9 and 3.1 eV emerged from modified samples through band tailing, also it is suggested that a heterojunction was formed in modified samples [3].
Acknowledgements:The present work was only made possible by FAP-DF, GRACO, University of Brasília, and my advisor Dr. Alysson Martins.References:[1] Brody, P. S. High voltage photovoltaic effect in barium titanate and lead titanate-lead zirconate ceramics. Journal of Solid State Chemistry, 12 (1975)[2] Osoro, G. M., Bregiroux. Structural and piezoelectric properties evolution induced by cobalt doping and cobalt/niobium co-doping in BaTiO3. Materials Letters, 166 (2016)[3] Shafir, O., Yang, J. Ferroelectric barium titanate derivatives containing Mo and Mg for transparent photovoltaic applications. Journal of Applied Physics, 126, (2019)

Free-standing ZnO nanowire/graphite membranes: time resolved photoluminescence

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ZnO nanowires (NWs) are being studied due to their promising applications in e.g. photonics, optoelectronics, solar cells, sensors and actuators. A few years ago, free standing membranes consisting of entangled ZnO NWs were developed by some of the authors on seed-free compacted graphite flakes [1]. The excellent UV emission of this hybrid material as probed by photoluminescence (PL) experiments and their free-standing nature gave clear indication that these membranes are very interesting for several technological applications. However, the origin of such large UV emission remained an open question. In this work, we study the emission of free standing ZnO/graphite membranes through time resolved and temperature dependent PL measurements. The samples were fabricated by vapor transport at high temperature in a tubular furnace under controlled (Ar+O_2) atmosphere. The NWs grew on catalyst-free graphite flakes that had been mechanically compacted within an alumina boat crucible. The self-supported membranes detached from the crucible base by exfoliation of the graphite substrate following thermal contraction during cooling. The PL time decay was analyzed in terms of decaying functions and time constants. A long lifetime component in a biexponential PL decay was inferred, which seems to be behind the large UV emissions detected at room temperature. Temperature dependent PL measurements reveal relatively low contributions from nonradiative recombination channels at room temperature, and a fine structure due to complexes emitting in the UV. The overall analysis indicates a low density of nonradiative recombination centers resulting from low precursor velocities achieved through a special geometry used during the vapor transport synthesis.

Acknowledgements: PICT2019-03795 (FONCyT, Agencia I+D+I), PIP970 and PUE08 (CONICET).

References:

[1] E. Tosi, O. Marin, M. Tirado, D. Comedi, Materials Letters vol. 269, 127658 (2020).

Improvement of the efficiency of Si-based solar cells by applying thin films of downconversion materials

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Climate change demonstrates the need to deploy renewable and CO_2-free energy in the coming decades. Solar energy, in particular photovoltaics, can play an important role in providing carbon-free energy [1]. The market for photovoltaic technologies is mostly dominated by silicon-based cells, corresponding to 90%. Silicon offers good stability and is the second most abundant element in the Earth's crust. However, the efficiency of silicon-based cells is limited by several factors, such as the intrinsic ones: transmission and thermalization, and the electronic ones: contact voltage, recombination, interface junction, and reflection. In this work we address thermalization losses, where the downconversion mechanism is proposed to overcome this problem. Therefore, the objective of this work is to study materials with the property of converting high energy photons into low energy photons. Lanthanides are good candidates to extend the functioning of silicon-based cells into the ultraviolet region, thus, europium (Eu) was selected as the downconversor agent to be used [2]. Starting from Eu chloride, different syntheses were tested, such as hydrothermal in the production of nanoparticles, for example. The synthed materials were deposited as thin films by spin coating and spray, the structural properties of the films were studied by XRD, the optoelectronic properties by PL, UV-vis transmission, and FTIR, and the morphological characteristics by AFM and SEM, with subsequent application of the films in the silicon cells and evaluation of the performance through the IPCE measurement.

Acknowledgements:

BYD Energy Brazil, FAPESP and CNPq.

References:

[1] Feltrin, A.; and Freundlich, A. "Material considerations for terawatt level deployment of photovoltaics." Renewable energy 33.2 (2008).

[2] Chen, C,; Zheng, S.; and Song, H. "Photon management to reduce energy loss in perovskite solar cells." Chemical Society Reviews 50.12 (2021).

Influence of Al/P ratio on the luminescence propertiesof erbium-doped alumino-phosphoro-silicate glasses obtained by sol-gel method

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Optical materials doped with rare earth ions (RE) are the backbone of telecommunications systems [1]. In this regard, aluminophosphosilicate (APS) glasses are widely adopted for active optical devices due to the higher solubility of RE ions compared to pure silica matrix [2]. These materials are typically prepared by the melt-quenching method, the sol-gel method is an alternative synthesis route allowing the formation of glasses at relatively low temperature, under ambient atmosphere and green solvents, as well as the control of the concentration of phosphorus and aluminum incorporated. [3]. In this work, we investigated the luminescence at 1.53 μm from erbium-doped APS glasses with different Al/P ratios (0.6, 1.0 and 1.5) by exciting the samples at 980 nm. It was verified that the Al/P ratio equal to 1.0 and 1.5 resulted in higher emission intensity and lifetime values compared to the 0.6 ratio. We further investigated the dependence of the luminesce with Er3+ concentration in the range of 0.05% up to 0.5% mol, and our results show luminescence quenching at concentrations higher than 0.2 (mol.%), indicating clustering, and decreasing the lifetime, and intensity emission. Nonetheless, lifetime values were measured on the order of 1 ms due to the presence of OH groups in the glass caused by the sol-gel process. In order to eliminate theses OH groups, the materials were densified under vacuum at 900 °C, resultingin an enhanced lifetime. Acknowledgements: CAPES, FAPESP, Canadian Excellence Research Chair program (CERC) in Photonics Innovations, Natural Sciences and Engineering Research Council of Canada (NSERC).References:[1] J. Ballato et al., Fibers 5.,1-25 (2017).[2] C. Shao et al., Opt. Mater. Express 10., 1169-1181 (2020).[3] R. Gvishi and I.Sokolov, J Sol-Gel Sci. Technol. 9., 635-648 (2020).

In situ UV generation by upconversion nanoparticles in photopolymerizable nanocomposites for disinfection of3D printing surfaces

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This work reports the synthesis and characterization of upconversion luminescent nanoparticles (UCNPs) of different fluoride matrices (CaF_2, LaF_3, Ca_2LaF_7) doped by the Yb^{3+}/Tm^{3+}pair, obtained by hydrothermal route, with the main objective of generating ultraviolet (UV) radiation in situ for decontamination purposes. To achieve higher UV emission intensity ($^1D_2 \rightarrow {}^3H_6$), a correlation was made between the concentrations of the Yb^{3+}/Tm^{3+}ratio and the IR→UV upconversion process, based on the Addition of Photon by Energy Transfer (APTE) mechanisms (Auzel Effect) between Yb^{3+}-Tm^{3+}ions and the cross-relaxation process between Tm^{3+}-Tm^{3+}ions [1-2]. A study of Fe^{3+}ions as a dopant showed that it contributed significantly to the IR→UV UC upconversion process. The obtained UCNPs had an average of 30 nm, presented in two crystalline phases: cubic (CaF_2) and hexagonal (LaF_3). The synthesized UCNPs were incorporated into a photopolymerizable resin for 3D printing and, as a result, a photopolymerizable UC nanocomposite (FPUCNC) was produced, which was subsequently printed in the shape of disks using mask stereolithography 3D printers (MSLA). Strategies are being established to increase the intensity of the UV emission as well as new mechanisms for Vis→UV UC instead of IR→UV UC to move excitation into the visible region. Microbiological tests to evaluate UV decontamination [3] generated in situ by UCNPs and FPUCNCs are being performed in collaboration with FIOCRUZ-PE using Zika as a model virus in a level-2 biosafety cabinet.

Acknowledgments:

This work was supported by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) RIMA Project - Edital Capes 09/2020 and CNPq Brazilian Agencies.

References:

[1] F. Auzel, J. Lumin. 45, 341-345 (1990)
[2] F. Guinhos, P. Nóbrega and P. Santa-Cruz, J. Alloys Compd. 323-324, 358-361 (2001)
[3] K. L. Nelson et al., Environ. Sci. Process Impacts 20(8), 1089-1122 (2018)

Magneto-optical sensors: Design and Development

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Magneto-optical materials have many applications in telecommunication systems, as Faraday isolators and rotators, can be used as sensors for both electrical and magnetical signals and have call attention for a variety of other fields such as those related to the monitoring of biosignals, including, but not limited to heart, muscles and brain. Despite the development of several devices based on single crystals, rare earths doped silica-based fibers and thin films, other challenges have emerged when the necessity of multifunctional devices with high sensibility is required. In this sense, the development of new glass compositions, suitable to produce more powerful devices, such as optical fibers, urges as the possible solution for an emerging market. In this work, we will show the study of promising glass compositions, the design of devices (optical fibers and discrete current sensors), as well as the trends in this multidisciplinary research field. The authors would like to acknowledge FAPESP (grant numbers 2012/07793-6) and SISFOTON-MCTi (CNPq-440217/2021-0) for financial support.

Nd^{3+}-Yb^{3+}:YAG thermal sensing for in vivo experiments:a comparison between materials synthesized by modified Pechini and solvothermal methods

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Nd^{3+}-YAG nanocrystals (NCs), known to exhibit two thermal couple levels from the $^4F_{3/2}$ excited state of Nd^{3+}around 940 nm, are currently evaluated as potential candidates for luminescent thermal probes within the first biological window BW-I, aiming biological applications. By luminescence intensity ratio approach, a relative thermal sensing (S_r) of 0.15 %·$K^{-1}$was determined in phantom tissues for detection and controlled thermal biomedical procedures [1], while Sr = 0.20 %·$K^{-1}$was obtained for NCs functionalized with a copolymer (PAA-b-PAM) [2]. Although the thermal sensing capacity of Nd^{3+}-YAG NCs, it is necessary to develop strategies to enhance the $S_r$value in order to reduce the thermal resolution (δ) value resulting in more precise temperature measurements. Therefore, we synthesized YAG co-doped with 0.25 mol% $Nd^{3+}$and varying from 0.10 to 2.00 mol% Yb^{3+}. In this work, we were able to increase three times the thermal performance of YAG nanocrystals thanks to the co-doping of YAG host matrix with different Ln^{3+}ions, in which we determined Sr = 0.67 %·$K^{-1}$and δ = 0.3 K. Our nanoprobes operate within the BW-II, with emissions at 1030 and 1064 nm. Moreover, we investigated the differences in the luminescent properties of these nanocrystals using two synthesis procedures, modified Pechini and solvothermal methods. In one hand, the first method allowed a facile route to produce nanocrystals and screening the Nd^{3+}-Yb^{3+}:YAG properties. On the other hand, the solvothermal allowed to obtain individual nanocrystals very well dispersed with morphology and size of 60 nm suitable for in vivo applications.

Acknowledgements:Centre national de la recherche scientifique (CNRS), Institut Néel, CAPES - Finance Code 001, CNPq and FAPEG.

References:

[1] A. Benayas, et al., Adv. Opt. Mater., 3, 687-694, 2015.
[2] A. Cantarano, et al., ACS Appl. Mater. Interf., 12, 51273-84, 2020.

Nonlinear optical properties of colloidal perovskite nanoplatelets superlattices

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All-inorganic lead halide perovskite nanoplatelets (PNPLs) have been explored in the last years because of their attractive optoelectronic properties such as high photoluminescence quantum yield and linear absorption coefficient [1]. Recently, we have shown that it is possible to form PNPLs superlattices formed by colloidal dispersion with viscous solvents such as Hexadecane [2]. In this work, we explore how the organization of PNPLs in superlattices influences their nonlinear refraction and two-Photon absorption (2PA) response. The study is performed in two PNPLs systems, two- and three-monolayer PNPLs, dispersed in hexane (C6, with viscosity coefficient at room temperature 0.31) and hexadecane (C16, with viscosity coefficient at room temperature 3.45). Our results indicate that 2PA cross section is six-fold enhanced when PNPLs are dispersed in C16, forming superlattices, compared to the analogs in C6, independently of the PNPLs thickness. It is still unclear the physical mechanism responsible for the enhancement of 2PA response for PNPLs organized in superlattice, however, one can speculate that this assembly of PNPLs breaks some of the selection rules for 2PA transition, increasing the density of allowed final states. Therefore, PNPLs superlattices emerge as an attractive alternative to increase 2PA cross sections in semiconductor nanomaterials.

Acknowledgements: We thank the Sao Paulo Research Foundation (FAPESP), National Council for Scientific Research (CNPq), and the Coordination for the Improvement of Higher Education Personnel (CAPES) for the financial support.

References:

[1] He T, Li J. Adv. Opt. Mater. Vol 6. (2018)

[2] Moral R.F, Malfatti-Gasperini A.A. ChemRxiv. https://doi.org/10.26434/chemrxiv-2022-38r3g (2022).

Optical and structural properties of heterojunctions based on MPA-capped silver indium sulfide QDs

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High quantum yield values, functionalizable surface and good light absorption efficiency are characteristics that make quantum dots promising materials for optical applications.[1] To improve performance, the production of heterojunctions favors the electron transfer process, increasing the efficiency of the reaction at the interface by decreasing recombination centers.[2] In this work, the optical and observed properties of quantum dots of indium silver sulfides (AIS) stabilized by 3-mercatopropionic acid and co-stabilized by citric acid were evaluated. From the modulation of the Ag^{+}/In^{3+}ratio, the modulation of theemission spectra from 625 nm (0.07) to 675 nm (0.5) was verified, with quantum yields (QY)decreasing from 5.0% to 3.9%, respectively. Coatings with ZnS and CdS led tohypsochromic emission shifts and different QY% regimes, starting from AIS (0.18) with a progressive increase in QY%, from 9.0% (1:0.25) to 10.4% (1:2). Coatings with CdS showedan increase of QY%, up to 6.8% and 5.6% (1:0.25 and 1:0.5) and followed by a decrease inthe quantum yield up to 3.4% (1:2). Both regimes are associated with growth mechanisms of nanostructures, via ionic exchange, controlled by polarizability of d^{10}ions. Leading to substitution processes of $Cd^{2+}$and Zn^{2+}in Ag^{2+}positions, since they are soft acids and Pearson's border, but which lead to different regimes of core@shell and alloy structures. The photoluminescence lifetime decay curves showed that ZnS and CdS coatings increased the mean lifetime from 251 ns to mean values of 280 ns, but increased the relative abundance of excitonic processes in the luminescence. The coatings improved the charge transfer dynamics on the surface, directly implying a greater photoluminescent response.

1 Kim et al. ChemSusChem 2022, e2022019252.

2 Sousa et al. Mat. Tod. Chem. 2020, 100238.

Optimization of luminescence intensity of gluthathione capped $AgIn_5S_8$@ZnS quantum dot alloys in aqueous medium

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New synthetic methodologies of quantum dots (QDs) look for simplicity, low cost and the non-use of toxic reducing agents have been the object of study in recent years.[1] Ternary nanocrystals, such as indium silver sulfide ($AgIn_5S_8$) stand out for their low toxicity when compared to systems based on cadmium and lead. In this work, an efficient route of synthesis via electrochemistry with a green character was used for $AgIn_5S_8$nanoparticles stabilized by L-glutathione, modulating the optical properties of light absorption and emission through different pHs and heat treatment.[2] It was observed that the nanocrystals obtained in the proportion 1:8, thermally treated for 60 minutes and at pH 7 showed better results of luminescence intensity, with a quantum yield of 3.1%. With the pH change, it was observed the displacement of the absorption bands and modification in the growth rate, as well as in the stability of the nanocrystal. To maximize the intensity, a coating with ZnS seeds electrochemically synthed and stabilized by GSH was carried out, with subsequent thermal treatment of the nanocrystals. The best results were from the use of the molar ratio of 1:1 between $AgIn_5S_8$and ZnS, forming nanocrystals with QY equal to 11.1%. The nanocrystals have a cubic crystalline structure, with peaks at 27.5, 46.3 and 52.7° for $AgIn_5S_8$, while they have peaks at 28.1, 47.4 and 55.4°. This 2θ displacement demonstrates the reduction of lattice parameters, resulting from the insertion of Zn^{2+}ions in the crystalline structure of $AgIn_5S_8$QDs in the position of Ag^+ions, forming the $AgIn_5S_8$@ZnS alloy. The results demonstrate a simple way of producing $AgIn_5S_8$@ZnS QDs with excellent stability and biocompatibility for respective biological applications without the need for further purification.

[1] (a) Jańczewski, D. et al. Eur. Polym. J., 54, 87, 2014.
[2] F. L. N. Sousa et al., Mater. Today Chem., 16, 100238, 2020.

Photochromic Behavior of Cationic NaphthaleneDiimides

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We will report in this presentation on the outstanding photochromic properties of naphthalene diimide (**NDI**) derivative bearing quaternary ammonium group (**DaO**). We found that a photoirradiation (λ = 365 nm) of **DaO** solutions triggers a fast color change, from colorless to dark red, that is not attributed to the photo reduction of the **NDI** core[1,2]. The observed phenomenon is accentuated for concentrations higher than 2 10^{-4} M and it is observed in different solvents such as THF, acetonitrile and water. We will show in this presentation that the photochromic behavior of **DaO** can be ascribed to a light-induced aggregation which leads to a significant modification of the electronic transitions resulting from the formation of H-type aggregates. In particular, our studies have revealed that these light-triggered self-assemblies can be dissociated by increasing the temperature to 70°C orby adding suitable salts (like $NaBF_4$). The reversible formation/dissociation of these assemblies was studied during irradiation/heating cycles followed by ^{1}H NMR and UV-vis spectroscopy measurements. Our results also support the conclusion that singlet oxygen is formed in solution when irradiation is performed under aerated conditions, which contrasts with previous reports demonstrating that aggregation usually decreases the sensitization yield.

Acknowledgements:
This work was supported by the São Paulo Research Foundation (FAPESP Grant n° 2021/06753-7, n° 2022/02711-0, n° 2022/06346-5 and n° 2021/01509-0). TBF also thanks the Ecole Normale Supérieure de Lyon (France) and USP.

References:
[1] MORAES, Thaisa BF et al. Journal of Luminescence, v. 204, p. 685-691, 2018.
[2] GONZALEZ, Laura et al. Communications Chemistry, v. 1(1) p. 77, 2018.

Photonic inorganic materials containing lanthanides for random lasers, luminescence nanothermometry and optical waveguides

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It will be present interesting results of oxide nanomaterials doped and co-doped with lanthanides presenting emissions on visible and near infrared regions. The materials studied were prepared by soft-chemistry methods (Sol-gel process and polymeric precursors method) of the YBO_3, $YAl_3(BO_3)_4$, $Al_4B_2O_9$, $Y_2Ge_2O_7$, $Gd_2Ge_2O_7$, $Y_2Ba_3B_4O_{12}$, beta-BaB_2O_4, $Y_4Al_2O_9$, and AMPYZCAD, containing Er, Eu, Nd, Tb, Tm, and/or Yb for random lasers, lighting devices, displays, nanothermometry and/or optical waveguides applications. We characterized and analyzed the structural, microstructural, and spectroscopic properties, by using X-ray diffraction (XRD), high-resolution transmission electron microscopy (HR-TEM), selected area electron diffraction (SAED), diffuse reflectance spectroscopy, Raman spectroscopy, photoluminescence emission spectroscopy and lifetime measurements techniques. In summary, $YAl_3(BO_3)_4$ and YBO_3 doped with Nd can be used as efficient and stable random lasers operating in the infrared regions (mainly around 1064 nm); amorphous thin films of $YAl_3(BO_3)_4$ containing Er possess high quality for waveguides with good light coupling coefficient; YBO_3 and $Y_4Al_2O_9$ co-doped with Eu, Er, Nd, Tb, Tm present white light emission under excitation in the ultraviolet region, interesting for lighting and display device $Al_4B_2O_9$, $Y_2Ge_2O_7$, $Gd_2Ge_2O_7$, $Y_2Ba_3B_4O_{12}$ and beta-BaB_2O_4 doped with Nd or co-doped with Nd and Yb present good relative thermal sensitivity (S_r) values (higher than 0.23 %/K, with a maximum of 2.0 %/K, depending on the host composition and lanthanide concentration) being useful for nanothermometers in biological and opto-electronic system and AMPYZCAD have blue light emission with quantum yield around 80%, which could improve the efficiency of commercial LED's by coating with appropriate mixture of AMPYZCAD and PMMA. We acknowledge UFG-LabMic and CRTi for strctural results. This study was supported by CNPq, CAPES-Finance Code 001, FAPEG, FAPESP, FACEPE and I. Néel-CNRS-UGA.

Sulfur quantum dots obtained by femtosecond laser ablation of elemental sulfur in water

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Sulfur quantum dots (SQDs) have been receiving great attention in recent years due its unique chemical, optical and biocompatible properties [1]. However, long reaction time, low photoluminescence quantum yield (PLQY), and very low S-to-SQDs conversion efficiency (lower than 1%) were reported as hindering to real applications [1,2]. Although, laser ablation is a known physical methodology applied to obtain nanoparticles of a lot of metals and oxides and to promote synthesis under unconventional conditions [3]. In this work, for the first time, a femtosecond laser was applied to synthe SQDs. For that, powder of elemental sulfur (S) was ablated in alkaline water solution containing PEG-400 through 1 h. Furthermore, the transparent brownish solution obtained was reacted with hydrogen peroxide solution at 3:4 ratio. As results, highest cyan emission was achieved using peroxide 3,75 %. The samples were analyzed by FTIR, Raman, TGA, XRD and luminescence measurements. Digital-image method was applied to determine the Hg concentration in environmental water.

Acknowledgments

This work was supported by: CNPq, CAPES and FACEPE.

References

[1] Gao, P., Huang, Z., Tan, J., Lv, G., Zhou, L. (2022). ACS Sustain. Chem. Eng., 10(14), 4634-4641.
[2] Shen, L., Wang, H., Liu, S., Bai, Z., Zhang, S., Zhang, X., Zhang, C. (2018). J. Am. Chem. Soc., 140(25), 7878-7884.
[3] Ferreira, A. F., Falcão, E. H. L., de Azevedo, W. M. (2023). Mater. Lett., 134365.

Temperature effects on the Raman spectrum of lead metasilicate glass, crystals and melt: vibrational anharmonicity and configurational transformation.

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The structural relaxation of glasses is directly related to the motion of atoms within the matrix, which is highly dependent on temperature. The motion of atoms may be either vibrational or translational. In the case of vibrational motion, the atoms vibrate around their equilibrium position with low amplitudes, and the configuration remains unchanged. On the other hand, in configurational relaxation, the atoms may diffuse from their equilibrium positions with increasing temperature. In this case, the timescale for configurational relaxation is much shorter than the experimental acquisition times, so time-averaged configurations are observed. In the present work, we will discuss and analyze in detail the results of the temperature effects on lead metasilicate, starting from the glass at room temperature up to 1220 K, 150 K above the melting point. The main experimental technique used was in-situ Raman scattering; some results were interpreted with the support of Molecular Dynamic Simulations.

The role of Eu^{2+}/Eu^{3+} ratio in Eu^{2+} based persistent phosphors explored by the case of $SrAl_2O_4$:EuDy

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The Eu^{2+}/Eu^{3+}ratio is a critical parameter that significantly affects the performance of Eu^{2+}-based persistent phosphors. Despite its importance, this ratio has often been overlooked, making it challenging to isolate other influences such as dopant concentration, particle size, or crystallinity. In this study, we aimed to provide valuable insights into the relationship between experimental conditions and the Eu^{2+}/Eu^{3+}ratio, as well as the luminescent properties of the $SrAl_2O_4$:EuDy phosphor. To achieve this goal, we produced ceramic samples under different atmospheric conditions and various concentrations. A low reducing atmosphere was mimicked by laser sintering, and a high reducing atmosphere was used by vacuum traditional sintering. We used X-ray diffraction, electron microscopy, X-ray absorption, and photoluminescence techniques to analyze the samples. Our results indicate that the dependence of the Eu^{2+}/Eu^{3+}ratio on dopant concentration is different under ambient and vacuum atmospheres. Specifically, the ratio exhibits a stronger dependence on concentration in the low reducing atmosphere compared to the high reducing atmosphere. Furthermore, we found that the ratio is a more relevant factor than the dopant concentration in determining the luminescent properties. Finally, we propose that the Eu^{2+}/Eu^{3+}ratio should be considered in conjunction with other factors to fully understand the luminescent properties of Eu^{2+}-based phosphors.

The role of intermolecular interactions in the luminescence of Eu^{III}-phosphor incorporated inpolymeric films

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The remarkable technological progress has led to a significant increase in the demand for optical materials, and tuning their photophysical features is crucial to enhance their luminescence, which can be done by combining spectroscopic and theoretical approaches [1]. Therefore, we present a theoretical protocol based on modelling the interfacial interactions in the composite based on the $SrY_2O_4:Ce^{IV/III}$(2at.%),Eu^{III}(3at.%) phosphor and the poly(methyl) methacrylate polymer (PMMA). All the calculations were performed using the GFN*n*-xTB code considering 50 monomers of the polymer around a 2×2×2 supercell ofthe phosphor, where eight Y^{III}sites were replaced by Eu^{III}. The simulation furnished information about the interactions, while their effect on the Eu-O bonds was obtained froma pseudo-diatomic model. SEM images revealed that increasing the phosphor quantity in the composite resulted in coarser films due to the agglomeration of the phosphor particles. Agglomerates were consequences of the polymer-phosphor intermolecular interactions, as illustrated from calculations, where Eu^{III}directly interacted with organic groups of the polymer. Decreasing the distance of the interacting atoms led to the strengthening of the intermolecular forces, affecting the photophysical features of the films. Stronger interactions promoted angular and bond length variations of the axial oxygens in the EuO_6polyhedra, leading to changes in the intensity parameters ($\Omega_{2,4}$). Hence, a computational methodology was developed to derive the influence of structural features on the photophysical aspects of the films in suitable accordance with the spectroscopic data. Accordingly, this protocol contributes to a better understanding of LnIII-based films, helping to bridge the gap to design strategies to enhance their performance.

Acknowledgements:
FAPESP (2022/08409-4), CNPq (309448/2021-2)

References:

[1] L. F. Saraiva, A. G. Bispo-Jr, S. A. M. Lima, J. Alloys and Comp. 938 (2023) 168595.

Two-photon absorption in colloidal nanomaterials

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Semiconductor colloidal nanocrystals (NCs) have emerged as a promising platform for applications that would benefit from large two-photon absorption (2PA) cross-section, as it has been predicted that quantum-confinement would enhance the oscillator strength for nonlinear optical transitions. However, after about two decades of intense study of 2PA in NCs, what are the lessons the community has learned from it? Here we will make an overview of the state-of-the-art of 2PA studies in quantum-confined semiconductors and show strategies to control the nonlinear absorption response of these nanomaterials. We show that, despite the fact that quantum confinement induced enhancement of 2PA in 3-dimensional confined semiconductors has not been demonstrated unambiguously yet, there are methods to obtain high 2PA response in nanomaterials by shape and heterostructure engineering. Using different types of core/shell heterostructure, we show that it is possible to decouple the magnitude of the nonlinear response from the emission wavelength, obtaining nearly one order of magnitude enhancement of 2PA cross-section in quasi-type-II CdSe/CdS core/shell, without shifting their emission wavelength. This can be useful for applications going from two-photon excited lasers to multi-photon excited 3D microscopy.

Biological temperature range using a optical thermometer based in $CsPbI_3$ perovskite and Er^{3+}

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The determination of temperature is a crucial parameter in numerous biomedical and industrial applications. In this context, the utilization of an optical thermometer presents a highly promising alternative to conventional thermometers, owing to its rapid response and non-contact capabilities. In the last decade, optical thermometry was studied using the luminescence of coupled levels of rare earth ions such as Er^{3+}. Moreover, the optical thermometers based on Er^{3+}have been used as a primary thermometer due its fluorescence intensity ratio responds linearly to changes in temperature mainly in the biological temperature range. In other hand, perovskite halide semiconductor nanocrystals ($CsPbX_3$, X = Br, Cl, I) present absorption and optical emission bands that are adjusted in the region of the visible spectrum (400 - 750 nm) [1]. These nanostructured materials have strong luminescence, and the absorption range is inserted in the first biological window. In this work, the glassy matrix doped with $CsPbI_3$and codoped with the Er^{3+}ion concentration varying in relation to the mass content of the glass matrix of 1 and 2% is being studied. The samples were characterized using Transmission Electron Microscopy/Electron Diffraction, Photoluminescence and UV-Vis Spectroscopy. This system is a primary thermometer, has a value of 4.3 K^{-1} to relative sensitivity at 298 K and shows to be an interesting candidate to an optical thermometer with application in the range of 298 to 363 K.

Acknowledgements:
The authors are grateful to the CNPq, CAPES-Finance Code 001, FAPEMIG and INCT/Institute of Photonics (INFO) for the financial support.

References:
[1] Franziska Krieg et al. Colloidal $CsPbX_3$(X = Cl, Br, I) Nanocrystals 2.0: Zwitterionic Capping Ligands for Improved Durability and Stability. ACS Energy Letters 2018 3 (3), 641-646.

Characterization of silica xerogel for plasmonicbiosensing

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We investigate the optical properties of systems comprised of functionalized gold nanoislands on the surface of silica xerogel monoliths for the development of biosensors based on Localized Surface Plasmon Resonance (LSPR). LSPR is highly sensitive to changes in the nearby chemical environment, making it an ideal choice for biosensors. To carry out this study, we functionalized the xerogel surface with biotin to detect avidin in aqueous solution. The silica gels were obtained by two-step sol-gel process from TEOS:H2O:Ethanol solution, using HCl and NH4OH solutions as catalysts. The gels were then aged and dried at 700oC to create the xerogels. To form the gold nanoislands, a thin film of 20 nm Au was deposited on the top surfaces of the xerogels and subjected to heat treatment up to 600°C. The resulting Au@SiO2 xerogels exhibited a color change from blue to pink, characteristicof gold nanoparticles. The surface of the AuNP@SiO2 xerogels were then functionalized intwo steps with cysteamine (CA) and N-hydroxysuccinimide-biotin (NHSB) for the detectionof avidin in an aqueous solution. The functionalization process was monitored by UV-Vis optical absorbance for each step, and aqueous solutions with different concentrations of avidin were used to test detection and sensitivity. An average redshift of 52 nm was observed in the absorbance spectrum of all tested concentrations (10-6M, 10-7M, 10-8M,10-9M and 10-10M), indicating that this system is a promising structure for plasmonicbiosensor applications.

Acknowledgements:
CNPq , CAPES and FAPERJ

References:
[1] RAO, A. Venkateswara; BHAGAT, Sharad D. Synthesis and physical properties of TEOS-based silica aerogels prepared by two step (acid-base) sol-gel process. Solid State Sciences,6, 945(2004).
[2] LYU, Yanchao et al. The biotin-avidin interaction in biotinylated gold nanoparticles and the modulation of their aggregation. Nanomaterials, 11, 1559(2021).

Comparison of Copper, Silver, and Gold Plasmonic Nanoparticles Efficiency in SERS and SERRS active platforms

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Plasmonic nanoparticles have been widely studied due to their special optical properties. Particularly, the study of light interaction with metallic nanostructures is rapidly emerging in plasmonic devices that are associated with analytical applications such as Surface-Enhanced Raman spectroscopy (SERS). Herein, we synthesized copper, silver, and gold nanoparticles (CuNPs, AgNPs, and AuNPs) for comparison of enhanced-Raman signal efficiency, using Violet Crystal and Rodhamine 6G (VC and R6G) as probe molecules. We also evaluated the analytical performance using Levodopa (L-Dopa), with the SERS substrates fabricated from the self-assembly methodology (SAM). Silicon substrates were functionalized with (3-mercaptopropyl)trimethoxisilane (MPTMS) and (3-aminepropyl)trimethoxisilane (APTMS) for further metallic covalent interactions. Surface-enhanced Raman and Surface-Enhanced Raman Resonant Scattering (SERS and SERRS) were evaluated for the plasmonic platforms using different lasers (532, 633, and 785 nm) for the respective molecules. Effectiveness syntheses were confirmed by UV-vis spectroscopy with plasmon bands at 582, 479, and 531 nm, corroborated with STEM-SEM micrographs that displayed a good homogeneity distribution. Additionally, the high control size distribution of metallic nanoparticles was also checked with dynamic light spectroscopy (DLS) and inductively coupled plasm mass single particle. The SEM micrographs reveal that the SERS substrates have an excellent metallic deposition on the Si surface, with favorable hot spots coupling to detect trace levels of molecules. A detailed Raman mapping was achieved to detect VC, R6G, and L-Dopa in the 10^{-3}–10^{-9} mol L^{-1} concentration range, with good reproducibility and sensitivity obtained in the analytical curves.

Acknowledgments
This work was supported by FAPESP (Proc. 2022/02893-1, 2018/25207-0, 2014/50906-9) and CNPq (Proc. 465452/2014-0, 407591/2016-8).

Deconvolution of Photoluminescence Spectra and Electronic Transition in Carbon Dots Nanoparticlesfrom Microcrystalline Cellulose.

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Carbon dots (CDs) are nanomaterials with promising applications in several areas such as optoelectronics and biomedicine due to their low toxicity and strong luminescence intensity. Understanding the emission and bandwidth of the fluorescence spectrum is still a challengein the scientific community due to the dependence of emission on the excitation wavelength. In this work, the photoluminescence properties of carbon dots obtained from microcrystalline cellulose (MCC) were studied [1]. The optical properties were studied through measurements of photoluminescence (PL), photoluminescence excitation (PLE), and absorption. Systematic adjustments were made through Gaussian deconvolution of the PL spectrums and the coupling between electronic states was analyzed through PLE and optical absorption measurements. From the Gaussian adjustments, bandwidth values of ~25 nm and ~100nm were found for the emission associated with the core and the surface states respectively, these values are consistent with the bandwidth of the PLE spectra with tuned detection in these states. As evidenced by the results, there are contributions from the core and surface functional groups in the luminescence emission of CDs [2]. Furthermore, with recent analyzes that attribute the radiative emission of CDs only to the surface trap states, we observed that the core present emission and absorption of light for samples with specific pH and reaction times. These results should contribute to the understanding of the electronic transitions of CDs for applications related to the luminescent optical properties of these nanomaterials.

Determination of the band positioning of nanocrystalline semiconductors by cyclic voltammetry

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The determination of the band positioning of semiconductor nanocrystals is a fundamental step in the determination of possible applications and performances of the materials. In this sense, electroanalytical techniques are powerful alternative strategies to XPS/UPS analysis. In this work, we evaluated the band positioning from the d10 MS ion sulfide series (d10 M=Zn2+, Cd2+, Hg2+ ions) with zinc blend structure, through cyclic voltammetry. The quantum dots (QDs) were electrosynthed in a cavity electrochemical cell, through a chronopotentiometric procedure (30 mA, 12.5 C), forming in situ HS- ions, using L-glutathione as a stabilizer in a proportion (1 :3:6, HS-, M2+, GSH), provide the formation of ZnS, CdS and HgS nanocrystals.[1] Being heated for 30 min under reflux. From the absorption spectra and the construction of Tauc curves, the values of optical bandgap (Egopt) of the ZnS QDs (λabs = 325 nm, Egopt = 3.80 eV), CdS (λabs = 380 nm, Egopt = 2.65 eV) and HgS (λabs = 800 nm, Egopt = 1.54 eV). ZnS and CdS nanocrystals present optical emission (λem) at 442 and 490 nm, respectively. HgS in the near infrared region. Relative band positioning were determined by cyclic voltammetry, with a glassy carbon electrode as a working electrode, Pt as a counter electrode, and Ag/AgCl 3.5 M as a refstabilizer, For measurements at pH 4, the cathodic peaks -1.26 V ZnS, -1.15 V CdS, -0.98V HgS (V vs. Ag|AgCl) were verified, evidencing the influence of pH on the deprotonation ofthe stabilizer and making the interface accessible of quantum dots. The experimental datawere compared with those calculated using Mulliken''s absolute electronegativity,[3] and small deviations between the electrochemical method (cyclic voltammetry) were verified.

Acknowledgments:
MEC, MCTI, UFPE.

References:

1 Green Chem. 16 (6), 2014, 3247.
2 Catal. Sci. Technol., 2023,13, 2377-2384
3 J. Power Sources 506 (2021) 230165.

Development of phosphor-in-glass materials for LEDs applications: YAG:Eu^{3+}, YAG:Dy^{3+}, YAG:Sm^{3+} and YAG:Tb^{3+} with PKAL glass

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The phosphors-in-glass (PiG) finds applications in electronic devices for conventional lighting and in the high power automotive headlight industry [1,2]. This work developed PiGs materials from oxyfluoride phosphate glass system and TR-doped YAG (TR=Sm^{3+}, Eu^{3+}, Tb^{3+}or Dy^{3+}in content of 0,01 mol). The glass $42P_2O_5$-50KF-$8Al_2O_3$(PKAl) in mol% was synthesized by the conventional melt-quenching, in air atmosphere. The YAGs were synthesized by the sol-gel method. For the preparation of the PiGs an amount of x=10, 20 and 30 wt% of doped YAG was added to the glass matrix (100-x) by sintering (T = 420°C). The synthesized glass showed an amorphous characteristic verified by the XRD data and the phosphors showed a XRD pattern corresponding to the pure YAG phase. The PiGs analyzed by XRD showed peaks characteristic of crystalline materials of the YAG phase and an amorphous phase corresponding to PKAL glass. In the FTIR spectra, it was verified that the profiles of the samples of YAGs correspond to the FTIR spectra reported in the literature, as well as the synthesized glass and PiGs have characteristic bands of phosphate glasses. The excitation and luminescence spectra for doped samples showed characteristic bands of Sm^{3+}, Tb^{3+}, Dy^{3+}and Eu^{3+}ions. The increase in the luminescence intensity was proportional to the amount of YAG doped. These results show that doped PiG materials are good candidates for optical and photonic applications, with wide application possibilities.

Acknowledgements: We are thankful the CAPES, CNPQ, FAPEMA, PPGCM-UFMA and LEOF.

References:

[1] I.J. Lee, H.S. Noh, V. Elayappan, D. Kwon, Y.H. Kim, M.K. Sun, S.E. Kim, H. Lee. Ceram. Int. 49 (2023) 14505-14511. doi:10.1016/j.ceramint.2023.01.040. [2] E.K. Ji, Y.H. Song, M.J. Lee, D.H. Yoon. Mater. Lett. 157 (2015) 89-92. doi:10.1016/j.matlet.2015.05.092.

Effect of Calcium and Europium Doping on Optical, Structural and Morphological Properties of TiO2 Nanocrystals

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Titanium dioxide (TiO_2) is a material widely used in photocatalysis applications, due to its high photoactivity and low cost[1]. In order to tune the properties of nanocrystals, doping with metal and non-metallic ions is performed. Doping alters the band gap, structural phase and size of TiO_2nanocrystals (NCs) [2]. Therefore, in order to investigate the effect of type and concentration of doping on the properties of TiO2 NCs, in this work we synthesized and characterized pure and doped TiO_2nanocrystals with calcium and europium ions. Vibrational, structural, optical and morphological properties were investigated using Raman Spectroscopy, X-Ray Diffraction (XRD), Optical Absorption (AO) and Scanning Electron Microscopy (SEM) techniques. Raman and XRD diffractograms confirmed the formation of TiO_2nanocrystals in the anatase phase. In the AO spectra, characteristic absorption bands of $TiO_2$and dopants were observed, and with increasing concentration there was a variation in the band gap of the nanocrystals. In the SEM images confirmed the formation of nanocrystals. Therefore, in this work it was demonstrated how the type and concentration of ions alter the structural, optical and and morphological properties of TiO_2nanocrystals.

Acknowledgements: CNPQ, FAPEAL and CAPES

References:

(1) Khlyustova, A.; Sirotkin, N.; Kusova, T.; Kraev, A.; Titov, V.; Agafonov, A. Doped TiO2: The Effect of Doping Elements on Photocatalytic Activity. Mater. Adv. 2020, 1 (5), 1193-1201. https://doi.org/10.1039/d0ma00171f.
(2) Fadlallah, M. M.; Eckern, U. Electronic and Optical Properties of Metal-Doped TiO2nanotubes: Spintronic and Photocatalytic Applications. New J. Phys. 2020, 22 (9). https://doi.org/10.1088/1367-2630/abae87. properties of TiO_2nanocrystals.

EFFECT OF EU3+ CONCENTRATION AND HEAT TREATMENT ON THE STRUCTURAL AND OPTICAL PROPERTIES OF SODIUM TITANATE MATRIX TARGETING OPTICAL THERMOMETRY

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Luminescent thermometry continues to be an important area of research due to the growing demand for increasingly smaller temperature sensors, as in the case of nanothermometers [1], and that they are increasingly efficient, for example, with greater emission and relative thermal sensitivity [2]. Based on this, in this work we investigate the structural and luminescent properties of host matrices based on sodium titanate, more specifically, undoped and Eu3+ doped sodium trititanate (Na2Ti3O7) and sodium hexatitanate (Na2Ti6O13), with focus on their potential use as luminescent thermometers. The influencesof Eu3+ doping and heat treatment (HT) on the structural and luminescent properties ofsodium titanate matrices were investigated. As a result, we found that both the Eu3+ ion concentration and the variation in the HT temperature influenced the transformation of Na2Ti3O7 to Na2Ti6O13. In addition, the increase in concentration intensified the luminescence intensity. However, high HT temperatures favored the decrease in emissionintensity. The sodium titanate matrix doped with 5% and thermally treated at 500 ºC wasthe one that presented the highest luminescence, and we found that the maximum relativethermal sensitivity obtained was approximately 0.34%C-1 at 25 °C, which is a value comparable to other luminescent materials presented in previous literature. Thus, we demonstrate that this material, specifically sodium hexatitanate doped with 5.0 mol% of Eu3+, is a potential candidate for luminescent thermometry.

[1] D. Jaque and F. Vetrone, Luminescence nanothermometry, Nanoscale 4(15), 4301 (2012).

[2] M. Quintanilla, E. Cantelar, F. Cussó, M. Villegas, and A. C. Caballero, Temperature Sensing with Up-Converting Submicron-Sized LiNbO3 :Er3+ /Yb3+ Particles, Applied Physics Express 4(2), 022601 (2011).

Effect of Nb_2O_5 concentration on the thermo-optical and structural properties of PANK glass matrix nanostructured with CdS semiconductor nanocrystalsand doped with Nd_2O_3

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Rare earth (RE^{3+}) ions doped glasses matrix are materials of high technological importance. Lately, these materials have been used as photonic devices, optical display devices, optical amplifiers, optical temperature sensors, optical thermometry, photovoltaic cells, NIR and visible lasers, nonlinear optical devices, etc. Particularly, in this work the thermo-optical and structural properties of the phosphate glass matrix (PANK) doped with $Nd^{3+}$with different concentrations of niobium oxide (Nb_2O_5) are being studied. This system was synthesized using the traditional melt cooling technique and are being characterized by analysis structural (X-Ray Diffractometer and Raman spectroscopy), optical (UV-Vis absorption spectroscopy and Photoluminescence) and thermal (Differential Scanning Calorimetry). The results show that the addition of Nb_2O_5in the phosphate glass matrix produces structural changes that alter the optical properties of the material, such as the increase of the refractive index and increase in the photoluminescence of rare earth ions. Thus, these glasses can be potential candidates for applications in photonic devices, such as: filters for UV wavelength, UV and IV band energy converters for visible, and in optical fibers in the third telecommunications window [1].

Acknowledgements:
The authors are grateful to the CNPq, CAPES-Finance Code 001, FAPEMIG and INCT/Institute of Photonics (INFO) for the financial support.

References:
[1] José Henrique Faleiro, Noelio O. Dantas, Anielle C.A. Silva, Helliomar P. Barbosa, Bruno H.S.T. da Silva, Karmel de O. Lima, Guilherme de Freitas Silva, Rogéria Rocha Gonçalves, Rodrigo Falci, Younès Messadeq, Isabela Dias de Oliveira Branco, Bianca M. Cerrutti, Henrik Bradtmüller, Hellmut Eckert, Jefferson Luis Ferrari, Niobium incorporation into rare-earth doped aluminophosphate glasses: Structural characterization, optical properties, and luminescence, Journal of Non-Crystalline Solids, Volume 605, 122173, 2023.

Effect of polarizability binary metal chalcogenides on optical and structures of I-III-VI quantum dots

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Ternary quantum dots I-III-VI2 or I-III5-VI8 have a luminescence dynamic associated with the scale and easy modulation via chemical composition, allowing extensive band engineering work. This work presents the modulation of the optical properties of glutathione-capped silver indium sulfide system (AIS QDs) when coated with binary nuclei of chalcogenides (MX, where $M = Ag^{+}$, Zn^{2+}, and In^{3+}; $X = S^{2-}$ and Se^{2-}). Evaluating the influence of the polarizability of cations and anions for the formation of heterojunctions or alloys, according to Pearson''s hard and soft acids and bases theory.1 Heterojunction formation was evaluated via the AIS/MX ratio, with respect to chalcogenides ($S^{2-}:X^{2-}$= 1:0.25, 1:0.5, 1:1, 1:2). QDs were characterized optically and structurally. The absorption spectra and Tauc curves showed trends regarding MS coatings: with small proportions of MS, hypsochromic shifts were verified, associated with the substitution of cations forming acceptor states ($Zn^{2+}_{Ag^{+}}*$) and donor states ($Ag^{+}_{In^{3+}}{}'$ and $Zn^{2+}_{In^{3+}}{}'$) By increasing the MS content (Ag^{+}, Zn^{2+}, and In^{3+}), bathochromic shifts are associated with the growth of nanoparticles. For In_2S_3, a decrease in quantum yield (R6G as standard) was verified. For MSe coatings, all samples showed a progressive decrease in quantum yields (QY%) from 29.2% for AIS to 17.8% (ZnSe), 11.1% (Ag_2Se), and 10.7% (In_2Se_3) ($S^{2-}:X^{2-}$= 1:0.5). MS coating led to QY% increase of 48.1% (ZnS), 34.3% (Ag_2S), and 30.3% (In_2S_3). The optical effects were more significant considering the interaction with Ag^{+} and Zn^{2+} ions, soft and border acids, respectively. These results allow us to single out trends and dedicate specific methods for engineering ternary semiconductor bands in water.

1 Mat. Today Chem. 16 (2020) 100238

Efficiency enhancement of silicon solar cell covered with GPA glasses doped with Eu3+ and Tb3+ ions andsilver nanoparticles

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The optimization of solar cell performance remains a fundamental goal of the photovoltaic industry. An effective approach to achieve this objective involves managing solar spectrum radiation on the solar cell surface by covering it with photonic glasses [1]. Glasses doped with rare- earth ions have shown to be candidates to manage the solar cell spectrum for photovoltaic devices. In particular, Alumina-lead-germanate (GPA) glasses have demonstrated potential for photonic applications such as random lasers, multijunction solar cells and waveguides [1-3]. Here, the electrical response of a silicon solar cell covered with GPA glasses is investigated. Samples of GPA glasses doped with Eu3+ and Tb3+ ions and silver nanoparticles were fabricated using the melt-quenching technique [1]. Moreover, samples prepared without these nanoparticles were also prepared and used as reference. The current-voltage (I-V) curves of silicon solar cells covered with GPA glasses were obtained, and then, critical performance parameters, such as short-circuit current (Isc), open-circuit voltage (Voc), Fill Factor, and efficiency, were determined. The results showed that the photovoltaic device efficiency is increased when compared with the bare solar cell.

References:

[1] Mattos, G. R. S., et al. Enhancement of multijunction solar cell efficiency using a cover layer of Eu3+, Tb3+ and Eu3+/Tb3+ doped GeO_2-PbO-Al_2O_3 glasses as spectral converter of solar radiation. Optical Materials 132, 112833 (2022).

[2] Câmara, J. G., et al. Random laser emission from neodymium doped alumina lead-germanate glass powder. Applied Optics 62, C59-C63 (2023).

[3] Camila, D. S., et al. Influence of Al_2O_3 on the photoluminescence and optical gain performance of Nd3+ doped germanate and tellurite glasses. Optical Materials 109, 110342 (2020).

Energy-looping and photon-avalanche-like phenomena in $Nd_xY_{1.00-x}Al_3(BO_3)_4$ powders excited at 1064 nm

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Recently, a photon-avalanche-like (PA-like) process based on trivalent neodymium ions (Nd^{3+}) under an unconventional excitation at 1064 nm was demonstrated using stoichiometric $NdAl_3(BO_3)_4$ particles. Although the Nd^{3+} can emit radiation at 1064 nm efficiently, they present very small absorption at this wavelength due to the absence of resonant ground-state transitions. However, phonon-assisted excitation ($^4I_{9/2} \rightarrow {}^4F_{3/2}$ and/or $^4I_{11/2} \rightarrow {}^4F_{3/2}$) followed by cross-relaxation between one excited ion ($^4F_{3/2}$) with another at the ground state ($^4I_{9/2}$), and subsequent phonon emissions {$^4I_{15/2}$, $^4I_{15/2} \rightarrow {}^4I_{13/2}$, $^4I1_{3/2} \rightarrow {}^4I_{11/2}$, $^4I_{11/2}$}, provide two ions at the $^4I_{11/2}$ state, from which can occur the resonant excited-state absorption to the $^4F_{3/2}$ level. Reestablishing the sequence of events, the absorption of photons at 1064 nm can increase greatly. Besides the emission around 880 nm ($^4F_{3/2} \rightarrow {}^4I_{9/2}$), there are thermal excitations to upper-lying states, with subsequent emissionsin the visible and near-infrared regions (480-2000 nm). Here, we investigate the role of theNd^{3+} content on the PA characteristics in $Nd_xY_{1.00-x}Al_3(BO_3)_4$ particles with x ranging from 0.05 to 1.00. It is known that the replacement of Y^{3+} by Nd^{3+} into the $YAl_3(BO_3)_4$ crystalline structure can introduce strong modifications of the lattice properties as well as in the photoluminescence characteristics, such as luminescence concentration quenching and broadening of spectral lines. Despite that, we observe, for low x (≤ 0.20) an energy-looping preceding the PA-like that ensues for $x \geq 0.40$. It is associated to the proximity among the Nd^{3+} ions, fundamental to the electric dipole-electric dipole interaction responsible for the Nd^{3+} energy transfer {$^4F_{3/2}$, $^4I_{9/2}$} → {$^4I_{15/2}$, $^4I_{15/2}$}. We discuss the present results focusing on emerging technologies with development of ultra-sensitive thermal sensors, and super-resolution imaging thanks to the giant nonlinearities in the input-output power dependences.

Enhancing Second and Third Harmonic Generation with MoS_2 Monolayers on Silicon Nitride Waveguides

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Silicon optical waveguides have excellent scalability and readiness, but their optical properties, such as two photon absorption (TPA) and absorption at visible frequencies, limit their potential for optical frequency conversion applications [1]. Silicon nitride (SiN) emerges as a viable alternative to silicon, with proven performance in both near IR and visible applications. However, SiN's low Kerr nonlinear coefficient and centro-symmetric properties offer limited efficiency for nonlinear frequency conversion. Integrated SiN photonics structures with 2D materials offer a promising route to enhance nonlinear opticalproperties of this material. Transition-metal dichalcogenides (TMDCs), such as MoS_2, have optoelectronic characteristics like direct bandgap, robust valley polarization, and significant electric tunability which can be useful for various applications [2]. Many TMDCs are inherently non-centrosymmetric, making them attractive for second-order nonlinear optical effects, indeed, WS_2, MoS_2, and $MoSe_2$ have been used for second- and third-harmonic generation [2, 3]. In this work, MoS_2 monolayers were transferred on top of SiN waveguides to explore optical frequency conversion. Our results show an increase in second and third harmonic production due to integration of MoS_2on the waveguide while pumping with high power laser of 150 fs duration, and 750 W peak power at 1560 nm.

Acknowledgments:
This work is supported by the São Paulo Research Foundation - FAPESP (grant nos. 2020/04686-8, 2022/07892-3, 2020/04374-6, and 2018/25339-4), CNPq, the Brazilian Nanocarbon Institute of Science and Technology (INCT/Nanocarbono), and MackPesquisa.

References:
[1] H. Chen et al., Light Sci. app. 6, e17060 (2017).
[2] V. Pelgrin, Y. Wang, J. Peltier, C. Alonso-Ramos, L. Vivien, Z. Sun, and E. Cassan, Opt. Lett. 47, 734-737 (2022).
[3] RI Woodward et al., 2D Mater. 4, 011006, (2017).

Eu^{3+}-doped SiO_2 –$Y2O_3$ -SnO_2 planar waveguides: refractive indexes and spectroscopic properties for application in optical sensors and amplifiers

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Recent modern applications in the field of photonics have implicated the use of waveguides, mainly due to the growing demand for optical sources and amplifiers operating at wavelengths compatible with fiber communication technology. [1] In this sense, the purpose of this research was to study the properties of Eu^{3+}-doped SiO_2–$Y2O_3$-SnO_2planar waveguides. The films, with Si/Sn/Y ratios of 70:15:15, were prepared by a sol-gel methodology and deposited by the dip-coating technique onto 10 μm SiO_2–Si (100) p-type silicon and Si (100) silicon. The growth of films was followed by specular reflectance, where it was possible to observe the dependence of the number of interference fringes with the quantity of deposited layers. From the number of fringes and the position of their maximums and minimums it is possible to estimate the index of refraction and film thickness at each deposition [2]. The optical characterization was accompanied by m-line, obtaining the number of modes and propagation angles, as well as the effective refractive index of eachmode at different wavelengths in polarization function (TE and TM). The thickness andrefractive index values were used to understand better the influence of temperature and annealing time on the densification of the bulk films. The refractive index shows the densityof the films, achieving relatively high light confinement within the waveguide structure, andnegligible optical loss due to surface scattering. The annealing temperature, 900 °C, atdifferent times (until the refractive index stabilizes), has been shown to be enough to promote the densification, pore elimination, and increase and control the refractive indexvalues.

Acknowledgements: This work was supported by CNPq, CAPES, FAPESP and INFO.

References:[1] FERRARI, Jefferson L.; LIMA, Karmel de O.; GONÇALVES, Rogéria R. ACS omega, v. 6, 2021.

[2] MANIFACIER, J. C.; GASIOT, J.; FILLARD, J. P. Journal of Physics E: Scientific Instruments, v. 9, 1976.

Gain clamping in random lasers

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Random lasers (RLs) rely on obtaining laser emission in disordered systems with gain [1]. The RL characterization is usually performed by the observations of bandwidth narrowing, increasing in the slope efficiency, time shortening of the upper level of the RL transition, intensity and spectral fluctuations, and even photon statistics transitions when increasing the optical gain. However, the gain clamping, which is a known phenomenon in conventional lasers, i.e. lasers with well-defined cavity, was never investigated in RL. Here we realize an experimental-theoretical investigation of the gain clamping in RLs, and demonstrate that it can be used as an alternative tool to characterize the transition from spontaneous emission to the RL regime. Two different RLs were investigated: one based on Nd3+ ions in crystalline nanoparticles [YAl3(BO3)4]; and another based on rhodamine 6G molecules and titanium dioxide (TiO2) nanoparticles as scatterers. Despite these systems present distinct features such as quantum yields and fluorescence lifetimes, the gain clamping was observed in both, and corroborated with numerical simulations based on a rate equation system for the atomic populations, and the energies at the random and a fluorescence emission, whose transitions departure from the same level. In both systems, at the onset of the RL emission, the fluorescence demonstrated a saturation behavior, evidencing the gain clamping. As a consequence, the fluorescence presents low fluctuation for excitation pulse energy larger than the threshold, on the other hand of the RL emission, which shows large fluctuations. We discuss our observations on the light of the statistical mechanics, with the observation of Gaussian or Lévy probability distributions for the intensity fluctuations.

[1] - A. S. Gomes et al. Recent Advances and Applications of Random Lasers and Random Fiber Lasers Prog Quantum Electron 78, 100343 (2021).

GOLD METALLIC NANOPARTICLES BIOSYNTHESIZED FROM EXTRACT OF HYMENAEA COURBARIL L. (JATOBÁ) ANTIBACTERIAL AND THERMO-OPTICAL PROPERTIES

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The green synthesis of metallic nanoparticles has been considered a promising and sustainable route due to the use of biological organisms, or parts of them that replace chemical reagents that may have a higher degree of toxicity [1]. Thus, phytochemical compounds from plant extracts are used as reducing and stabilizing agents to obtain metallic nanoparticles [2]. In this work, we synthesized gold nanoparticles (AuNPs) in different concentrations of gold solution (100 μL, 200 μL and 300 μL) through green synthesis using the extract of leaves of Hymenaea courbaril L. (Jatobá). The results showedsurface plasmon resonance (SPR) bands at 526 nm, indicating the formation of AuNPs. TheZeta potential analyses showed that the NPs surface has negative values of −17 mV, indicating good stability. The AuNPs have average hydrodynamic size ranging from 58 nmto 193 nm. The vibrational modes, characteristic of phytochemical compounds (flavonoids, terpenes and alcohols) present in Jatobá extract were observed by FTIR analysis. The thermo-optical properties were investigated by thermal spectroscopy, and showed values of thermal Diffusivity between 8x10−4 cm2/s to 10x10−4 cm2/s. The samples were tested for antibacterial activity with two different types of bacteria (gram positive and negative), and it was found that both NPS and Jatobá extract have antibacterial activity. These gold nanoparticles are promising material for future applications in nonlinear optical and health area.

Acknowledgements: FAPEMA, PPGCM, LIMAV, FISMAT, LBM and UFPI.

References: [1] BHARADWAJ, K. K. et al. Molecules, 26, 6389. 2021.
[2] Alizadeh, S. R. et al. Catalysts, 13, 63, 2023.

GRAPHENE SHEETS AND NANOCOPPER FILMS DEPOSITED ON SILICA SUBSTRATE: A MATERIAL FOR USE IN ELECTRO-OPTICAL DEVICES.

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The use of carbon nanomaterials, such as graphene, has become more interesting due to its fantastic properties, allowing several applications. The present work aimed the synthesis and characterization of mono/bimetallic gold or silver/copper NPs thin films with graphene sheets on silica substrate, in a single thermal synthesis step. For this, silica coverslips were cleaned and functionalized with (3-aminopropyl) triethoxysilane (APTES) together with the chosen metal and polystyrene (PS) as an extra carbon source and taken to a muffle furnace at 350 °C for 30 minutes. In the end of the synthesis steps, the films were characterized through UV-Vis spectroscopy, to verify their LSPR, X-ray diffraction (XRD) to verify the crystalline structure, thermal analysis and Raman spectroscopy (Raman) to confirm the synthesis of graphene and transmission electron microscopy (TEM) images to verify nanocopper shape, and the presence of 2D carbon materials. All films obtained with copper precursors showed signs of graphene sheets traces on TEM images. Thus, the use of PS as an extra carbon source resulted in a graphene amount increase with an environmentally friendly synthesis. Samples containing 1:100 of PS resulted in the TEM images a high amount of amorphous carbon mostly, in the 1:10 a mixture of amorphous material and graphene, and in the 1:1 samples showed mostly graphene. These data were confirmed through thermal and Raman analyzes for Cu and AgCu samples.

Acknowledgements:
INCT Nanocarbono, INCT NanoVida, GQM-UFPR, CME-UFPR, LAMAQ - UTFPR.

References:

PONNAMMA, D; et al. Composites Part B: Engineering, v. 204, p. 108493, 2021.

Growth of silver/nickel (Ag/Ni) bimetallic nanoparticlesin silica and FTO substrate.

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The development of bimetallic nanoparticles has been gaining prominence, due to their properties that may differ from the monometallic ones. Applications for these nanomaterials are, for example, in electronic circuits and solar cells. Synthesis routes that result in these materials deposited in substrates can facilitate their use in different devices. For this, our work aimed the synthesis and characterization of monometallic and bimetallic (silver and/or nickel) nanoparticles thin films on silica and FTO substrates, in a single synthesis step, witha thermal reduction of the metallic precursors. For this, after cleaning both substrates with HNO_3solution, they are rinsed with deionized water, and the substrates are dried with hot air and placed in solutions containing 3-aminopropyltriethoxysilane (APTES) in toluene (10% v/v) with the metals precursors ($AgNO_3$and/or $Ni(NO_3)_2$), remaining under 60 °C heating for3 hours. After that, the reduction of metallic ions was carried in a 300 °C muffle for 20 minutes, until complete the APTES pyrolysis and cations reduction processes. All different films were characterized by UV-Vis spectroscopy, to verify their Localized Surface Plasmon Resonance - LSPR, indicating the success of metallic and bimetallic films preparation by the proposed route. Transmission Electron Microscopy (TEM) images were performed to obtain information such as the nanoparticles average and shape in each sample, and to understand the structure of bimetallic films. Scanning Electron Microscopy (SEM) images, in progress, will allow analyze the homogeneity, composition and stability of the synthed films.

Acknowledgements:
INCT Nanocarbono, INCT NanoVida, GQM-UFPR, CME-UFPR, LAMAQ - UTFPR.

References:

PAJOR-ŚWIERZY, A. et al., Advances in Colloid and Interface Science, v. 299, n. October 2021, 2022.

Influence of Amine Concentration on theCrystallization of Naphthalic Imides

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1,8- naphthalimides and 1,4,5,8- naphthalenediimides, as supramolecular units, are used as fluorescent glow agents, intracellular biomarkers, and model compounds for photoinduced electron transfer [1]. In this study, the investigation of the effect of amine concentration on the imidization of 1,4,5,8- and 1,8-anhydrides was carried out, taking into account that the amine-imide interaction during the reaction may lead to the formation of dyes with new photophysical properties, without, however, there being structural modification of the aromatic imide. Thus, using reactions between anhydride and amine in different percentages, it was studied the effect of the amine concentration in the synthesis of imide, in terms of coloration and brightness. Then, the respective crystals of these imides were obtained using ethanol as solvent. For the characterization of the materials melting point determination, infrared, X-ray and fluorimetry analyses were performed. For melting point analysis, it was obtained an average temperature of 171.8°C and for fluorimetry analysis the imides presented peaks around 464 nm, varying only in the intensity values. For the infrared analysis, it was possible to confirm that there was a reaction of imide synthesis, and all the characteristic peaks of an imide were observed. Despite the similarities in the characterization results, some imides presented different colorations, as well as their respective crystals, even though the using of a same solvent. So, it is possible to infer that the percentages of amine used were responsible for this change in coloration presented by the studied imides and the best percentage of amine to be used is above 30%.

Acknowledgements: Grant n° 21/01509 and 2022/07533-3 (FAPESP)

References:[1] Niu, C. G; Yang, X; Lin, W. Q; Shen, G. L; Yu, R. Q. N- Allyl-4-(N-2' -hydroxyethyl)amino-1,8-naphthalimide as a fluorophore for optical chemosensing of nitrofurantoin. The royal Society of chemistry, analyst, 2002, 127. 512-517.

Influence of the energy transfer of CdSe nanocrystalson the gold nanorods properties

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Gold nanorods and CdSe nanocrystals are distinct types of nanomaterials with unique properties. Gold nanorods are elongated, rod-shaped particles composed of gold atoms, and find applications in various fields such as medical imaging, diagnostics, catalysis, and drug delivery. On the other hand, CdSe nanocrystals are spherical particles composed of cadmium selenide molecules, and are used in optoelectronic devices like solar cells, light-emitting diodes, and lasers. The energy transfer between CdSe nanocrystals and Au nanorods can occur through multiple mechanisms, including Förster resonance energy transfer (FRET), plasmon-mediated energy transfer, and exciton-plasmon coupling. FRET involves the transfer of energy between electronic excitations in two different molecules or nanostructures, typically mediated by dipole-dipole interactions. Plasmon-mediated energy transfer, on the other hand, is the transfer of energy between plasmons in two different nanostructures, such as CdSe nanocrystals and Au nanorods. In this work, three group of samples were studied. The first group are CdSe nanocrystals were synthetized using two different growing time to form different sizes of nanocrystals. The second group are the Au nanorods synthesized in order to obtain different aspect ratios using distinct $AgNO_3$concentrations during the synthesis. And the third group the mixture contends CdSe nanocrystals and Au nanorods. The samples have been characterized using photoluminescence and absorption spectra and thermal lens technique to determine the optical properties and the energy process transfer.

Acknowledgements:

The authors are grateful to the CNPq, CAPES-Finance Code 001, FAPEMIG and INCT/Institute of Photonics (INFO) for the financial support.

References:

[1] Huan Zong et al. Plasmon-Enhanced Fluorescence Resonance Energy Transfer. Chem. Rec., 19 (2019), pp. 818-842.

Internal energy transfer from nanocrystals to Co^{2+} ionsat Bi_2S_3 tetrahedral sites embedded in host glass

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We experimentally evidenced the internal energy transfer (IET) in Co-doped Bi_2S_3Nanocrystals (NCs) embedded in borosilicate host glass produced by the fusion method. Transmission electron microscopy images showed quantum dots in glass with an average size of 5.0 nm and interplanar distances of around d_{212}= 0.280 nm, which corresponds to the Bi_2S_3semiconductor. Energy dispersive X-ray spectroscopy analysis confirmed the chemical elements Bi, S and Co in the NCs. X-ray diffraction showed the peaks associated with the crystalline planes that are characteristic of the Bi_2S_3orthorhombic crystalline structure. The shift to greater angles of the (212) plane with increasing Co-content confirms doping in the orthorhombic unit cell. The changes in the optical absorption (OA) and photoluminescence (PL) spectra are strongly related to the tetrahedral (Th) symmetry of the Co^{2+}ions within the Bi_2S_3 NCs. The Racah and crystal field energy parameters confirm the symmetrical environment of the dopant ion. We clearly observe in the PL and OA spectra radiative and non-radiative IET from Bi_2S_3 NCs to Co^{2+} Th ion energy states. The valleys and peaks of the emission and absorption bands correspond exactly to the coordinate state of the $[CoS_4]^{6-}$ion. The PL and OA spectra reinforce the internal energy transfer process when analyzing the increased overlapping of the Bi_2S_3NCs' exciton bands and the decrease in stimulated emissions from Co transitions at Th sites [1].

Acknowledgements:
The authors gratefully acknowledge financial support from the following Brazilian agencies: MCT/CNPq (409359/2016-5), PDJ/CNPQ (150297/2022-0), Capes, Fapemig (APQ-00211-17), and Minas Gerais Chemistry Network (MG-CN), supported by Fapemig (project CEX-RED-00010-4).

References:
[1] E.V. Guimaraes, A.S. Silva, G.A. Azevedo, N.O. Dantas, R.S. Silva, Internal energy transfer from nanocrystals to Co2+ ions at Bi2S3 tetrahedral sites embedded in host glass, Journal of Luminescence 255 (2023) 119517.

Investigation of non-linear thermo-optical properties of gold and silver nanoparticles obtained through green synthesis from Anacardium occidentale (Cashew tree) leaf extract

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In this work, the thermo-optical properties of gold and silver nanoparticles (AuNPs, AgNPs) obtained through green synthesis from Anacardium occidentale leaf extract were investigated. The studies of the non-linear optical properties of these nanostructures are important due to the localized surface plasmon resonance (SPRL) these materials are promising for several applications, such as photoconductors, photothermal therapies, biosensors, and optical limiters [1-3]. The nanoparticles were obtained through the reduction of silver and a gold solution, using leaf extract of the Anacardium occidentale (Cashew Tree), the samples were investigated by changing the concentrations of AgNPs and AuNPs. Through the absorption spectra obtained from the UV-VIS, we found the SPRL bands at 460 and 525 nm for AgNPs and AuNPs respectively, in which indicates the formation of nanoparticles. The SPRL peaks decrease linearly with the reduction of the nanoparticle concentration. With the Zeta potential analysis, we identified that the surface of the nanoparticles has a negative charge of -17 mV, indicating good stability. The nanoparticles had hydrodynamic diameters of 110 and 193 nm for AgNPs and AuNPs correspondingly. The thermo-optical properties were investigated using thermal lens spectroscopy, the AgNPs and AuNPs nanoparticles presented thermal diffusivities of the order of $8,71 \times 10^{-4} cm^2/s$ and $6,32 \times 10^{-4} cm^2/s$ respectively, the studies show that the propertiessuch as thermal diffusivity is sensitive to the change in intensity of the SPRL peak. Usingthe z-scan we identified that both AgNPs and AuNPs nanoparticles presented a non-linearthermal response, with a non-linear refractive index of around $10^{-3} cm^2/W$. Nevertheless, theresults indicate that AuNPs and AgNPs nanoparticles are promising materials for applications in photothermal therapy.

Investigation the optical and structural properties ofMn-doped Sb_2S_3 nanocrystals embedded in host glass

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Dilute magnetic semiconductor (DMS) nanocrystals (NCs) xMn-doped Sb_2S_3embedded in host glass were synthesized by the fusion method. Transmission electron microscopy images show the formation of xMn-doped Sb_2S_3NCs with increasing size (2.1 to 3.8 nm) as a function of concentration (x = 0.00 to 0.40), respectively. Energy dispersive X-ray spectroscopy analyzes confirm the S, Sb and Mn precursor elements of DMS NCs. Atomic and magnetic force microscopy measurements show the magnetic contrast patterns resulting from the incorporation of Mn atoms into the structure of Sb_2S_3. The electronic paramagnetic resonance spectrum confirms the presence of Mn^{2+}ions ($3d^5$) located in the crystal field of the Sb_2S_3semiconductor due to six hyperfine transitions. The redshift of the Raman vibrational mode (^1A_g) with increasing xMn concentration gives strong evidence for the presence of Mn^{2+}ions in sulfur vacancies (VS) in the Sb_2S_3 unit cell. Optical absorption and photoluminescence (PL) spectroscopy measurements for xMn-doped Sb_2S_3NCs show the subtle tunable redshift of the band gap and excitonic recombination in the green-red region, with increasing x concentration. PL confirms that dopant Mn^{2+}ions fill V_{S2}that are dominantin Sb_2S_3. Therefore, the position of the energy states after the increasing concentration of Mn^{2+}dopant ions gives rise to a luminescence insight for a future scenario of research and applications involving sustainable DMS NCs [1].

Acknowledgements:
The authors gratefully acknowledge financial support from the following Brazilian agencies: MCT/CNPq (409359/2016-5), PDJ/CNPQ (150297/2022-0), Capes.

References:

[1] R.S. Silva, E.V. Guimaraes, N.F. Cano, R.H. Rosa, H.D. Mikhail, A.C.A. Silva, N.O. Dantas, Raman and EPR Characterization of Diluted Magnetic Semiconductor Sb2-xMnxS3 Nanocrystals Grown in a Glass Matrix, J. Phys. Chem. C 123 (2019) 5680-5685.

Lanthanum fluoride germanate glasses forphotosensitive applications

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A kind of glass that has drawn much attention in photonics is the photo-thermo-refractive (PTR) glass [1]. These are photosensitive materials that present changes in the refractive index after radiation and subsequent heat treatment, due to precipitation of nanocrystals in the glass matrix [2]. Normally, silicate glass is used, but its high vibrational energies limits its optical window in the infrared. An alternative to this problem is the use of compounds with lower phonon energy. Therefore, this work focused on the synthesis of a PTR glass with high LaF3 contents. To evaluate the influence of melting time and temperature on the samples, the melting of the composition 50GeO2-15K2O-35LaF3 was performed in three conditions: melting at 900°C for 1 hour and 1180°C for 30 minute melting at 900°C for 1 hour and 1180°C also for 1 hour; and melting of only the oxide for 6 hours at 1180°C and 1hour with the fluoride at the same temperature. No significant differences were noted in thethermal analysis, and the first condition was chosen. After the synthesis of glasses, thesamples were thermally analyzed by DSC and the presence of two crystallization peaks wasnoted in the nominal compositions 45GeO2-15K2O-40LaF3 and 45GeO2-10K2O-5NaF-40LaF3, being these samples the most promising for precipitation ofLaF3 nanocrystals. The next step consists of crystallization study by heat treatment. Silver will be used as the photosensitive agent for the growth of nanocrystals. Subsequently, thesamples will be irradiated with UV light, thermally treated and characterized in relation to the optical (UV-Visible and photoluminescence spectroscopy) and structural properties (XRD).

[1] L. Glebova, K. Chamma, J. Lumeau, and L. Glebov, “Photo-Thermo-Refractive glass - Properties and Applications,” pp. 1-3, 2011.

[2] O. M. Efimov, L. B. Glebov, L. N. Glebova, “High-efficiency Bragg gratings in photothermorefractive glass,” Appl. Opt., vol. 38, no 4 (1999).

LOW COST EQUIPMENT FOR DETERMINING THE EFFICIENCY OF PHOTOCATALYSTS

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In several works related to the production of paper, plastic, cosmetics, food, and others, photocatalysts are used to degrade, catalyze, protect, clean, acidify and sterilize. A photocatalyst is an agent that can change the rate of a chemical reaction when exposed to light. Several materials have photocatalytic activity, and indeed, many more are to be discovered in the future. In this context, it is understood that it is essential to know the characteristics of a photocatalyst, mainly regarding its speed and efficiency in a chemical reaction. The general objective of this work is the development of low-cost, versatile, portable, and cloud-integrated equipment for determining the efficiency of photocatalysts using the principles of photodegradation of various dyes. This project offers an easy-to-replicate solution for universities. In addition, the aim is to increase the accuracy of the calculations performed to obtain the photocatalyst degradation yield since they usually are at most a dozen points in a photocatalyst characterization graph. Through the proposed solution, using measurements at small sampling intervals, e.g., on the order of seconds, procedures that usually last hours or even days can be characterized in an automated way, with low cost and greater precision. An application for data acquisition is developed and embedded in a microcontroller module, the ESP32 platform, which in turn is coupled to communication, measurement, and actuation devices, such as LEDs, light sensors (TEMT6000), and fluid pumps. Web and Mobile applications were developed for communication with the ESP32 module and for recording, viewing, and analyzing measurements.

Luminescence Concentration Quenching in Nitrogen-Doped Graphene Quantum Dots

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The investigation of the luminescence mechanisms of carbon-based quantum dots has been a fruitful research subject in recent years [1,2]. However, even though the synthesis to obtain highly efficient graphene quantum dots (GQDs) with redshifted photoluminescence (PL) have been improved, few works have exploited the occurrence of luminescence concentration quenching (LCQ) in suspensions based in GQDs. This work aims to investigate the LCQ of graphene quantum dots doped with nitrogen (GQDN), synthetized from sucrose, in different solvents. The synthesis started with the carbonization of sucrose in sulfuric acid. Nitrogen doping was achieved by reflux in nitric acid. The resulting GQDN were dispersed in dimethylformamide (DMF), dimethyl sulfoxide (DMSO), isopropyl alcohol (C3H8O) and deionized water. GQDN dispersions in H2O and C3H8O presented PL in the blue region, when excited in the UV region (340-360 nm). On the other hand, GQDN dispersed in DMF and DMSO, showed a broad PL band (450-600 nm), when excited between 400 and 550 nm. The results also showed that in suspensions prepared with DMF and DMSO, LCQ occurred when GQDN concentrations exceeded 1.12 g/L and 2.65 g/L, respectively. In samples prepared in H2O and C3H8O, PL did not exhibit LCQ within the range of investigated concentrations. The results herein may be employed for the optimization of GQDN for applications in sensors, WLEDs, and random lasers.

Acknowledgements:
This work was supported by the National Institute of Photonics (INCT) program of CNPq and by CAPES for the master''s scholarship.

References:
[1] Rocha et al, Appl. Sci. 2022, 12(17), 8686.
[2] Thangadurai et al, Mat. Sci. Eng. B, 2022, 278, 115633

Luminescent films containing rare earth fluoride nanoparticles for application in packaging and smart labels and waveguides

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When doping systems with lanthanide ions, luminescent materials can be developed with interesting properties depending on their characteristics such as transparency at the excitation wavelength, the high solubility of lanthanide ions in the solid lattice, phonon energy, and chemical and thermal stability [1]. Our goal is the synthesis of redispersable luminescent Ln^{3+}-doped rare earth fluorides particles (Ln^{3+}= Eu^{3+}, Er^{3+}/$Yb^{3+}$and Tm^{3+}/Yb^{3+}) with controlled morphology and dimensions, and luminescent nanocomposite monoliths and films with potential application in photonics, as nanothermometers. Spectroscopic properties were studied, and up-converting luminescence was detected under excitation at 980 nm. In the emission spectra, bands are observed in the green (525 nm) and red (660 nm) regions, indicating that the materials have emissions in the yellowish-green region. The preparation of films is carried out from colloidal suspensions containing nanoparticles dispersed in the solvent and (bio)polymers, organic-inorganic and inorganic hybrid based on silica (by sol-gel methodology). The depositions are made on PLA filaments for 3D printing.

Acknowledgments:
This work was supported by CNPq, CAPES, FAPESP and INFO.

References:

[1] Dong H, Sun LD, Yan CH. Chem Soc Rev. 2015;44:1608-34

Luminescent, Linear and Second-Order Non Linear Optical properties of Sodium Tantalum Germanate Glasses

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Alkali tantalum germanate glasses are interesting optical materials with high refractive index, high transparency in the middle infrared and are efficient hosts for rare earth luminescence [1,2]. These glasses are characterized by a limited Ta2O5 solubility in the germanate network and formation of tantalum rich regions in the glass structure built from interconnected TaO6 and TaO7 units. Hence, the glass forming domain in the ternary system (90-x)GeO2-10M2O-xTa2O5 (M being an alkaline ion) is limited from x=0 to x=20 mole%. Another interesting point is that transparent glass ceramics containing tantalate bronze-like structures (Na2Ta8O21) can be obtained from the most Ta2O5 concentrated samples. Such Er3+-doped glasses and glass-ceramics exhibit improved luminescent properties in the infrared and tunable red and green upconversion emission depending of the composition and crystallization state. The detailed crystallization mechanisms were investigated in these glasses and it appeared that short heat-treatment times around the onset of crystallization promote the volume precipitation of perovskite Na2Ta8O21 whereas longer heat-treatments induce the formation of surface spherulites associated with the crystallization of quartz-like α-GeO2. This latter phase is associated with a strong SHG (Second Harmonic Generation) signal generated from the spherulites. Another efficient way of promoting SHG in these glasses is the thermal poling under high DC voltage, with the sodium depleted layer and related static electric field responsible for a strong SHG signal associated with EFISH mechanisms. The use of microstructured electrodes also appeared to be suitable for the microimprinting of optical patterns with microscaled controlled linear and non linear optical properties and topology.

Luminescent thin films based on Er³⁺-doped germane-tellurite nanoglasses

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In this work, thin films were fabricated by spin-coating at different speeds and their properties were studied using characterization techniques such as luminescence under 980 nm excitation, AFM thickness and texture measurements, and Raman spectroscopy. The first step was to nanostructure Er^{3+}-doped germane-tellurite glasses (the set of glasses has the chemical composition: 59.95% TeO_2- 20% ZnO - 10% GeO_2- 10% Na_2O - 0.5% Er_2O_3). Afterward, mix the nanoparticles with anisole (solvent) and finally with Polymethylmethacrylate (photoresist). Finally, the mixture was deposited on pure Si substrates using the spin-coating technique, where the influence of rotation speed on the thickness of the thin film was analyzed [1]. The results obtained revealed that the spin-coating speed influences the texture and thickness of the films, which in turn affects the optical and structural properties of the materials. Furthermore, strong light emission in the green region was observed in our thin films, suggesting its possible application in photonic devices and offering novel insight into the synthesis and characterization of thin films by spin-coating [2].

Acknowledgments: Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) - Finance Code 001, the São Paulo Research Foundation (FAPESP) under the grant 2013/07276-1, and Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) through the Centro de Pesquisas em Óptica e Fotônica (CePOF)–São Paulo–Brazil (process 314505/2021-0).

References:
[1] J.L. Clabel H et al., Phy. Chem. Chem. Phy. 22, 15022 (2020)
[2] G. Lozano et al., Sci Rep. 12, 5015 (2022)

Magneto-plasmonic hybrid Au-Fe(oxide) nanoparticles: synthesis and SERS applications

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Magneto-plasmonics (MP) refers to multifunctional nanomaterials that simultaneously exhibit plasmonic and magnetic phenomena. This blending of magnetism and photonics at the nanoscale is generally achieved by combining plasmonic materials such as Au and Ag with magnetic materials such as 3d metals and their oxides [1]. In this work, we present experimental results on the laser ablation synthesis in solution (LASiS) of Au-Fe (oxide) core-shell nanoparticles (NP) in water. Our methodology consists of two steps: first, a gold target is irradiated with 1064 nm (400 ns; 0.5 mJ) pulses from a fiber laser at 50 kHz, leading to pure Au second, a glass vessel filled with the Au NP colloidal solution prepared in the first step is further used for the laser ablation of an iron target. Different LASiS setups were tested, with varying Au and Fe target-ablation time therefore, optimal Au and Fe colloidal concentrations were determined to synthe very stable hybrid Au-Fe (oxide) nanoparticle solutions. NP characterization via UV-Vis spectrometry shows a surface plasmon band centered at 530 nm, which accounts for the solution's vivid purple color; in addition, DLS and TEM reveal spherical NP with an average of 10 nm. NP structure characterization through EDS and XPS indicate the presence of iron oxide, which explains why a small neodymium magnet placed at the bottom of a test tube filled with the colloidal solution can attract all the NP to the bottom in less than 5 minutes. These core-shell Au-Fe (oxide) NP are promising materials for biophotonic applications, including bioseparation, in vivo imaging, and sensing. As a proof-of-concept test, we successfully used our Au-Fe (oxide) NP as substrates in Surface Enhanced Raman Spectroscopy (SERS) experiments to detect minute amounts of biological molecules such as amino acids and urea in aqueous solutions.

[1] New Trends in Nanoparticle Magnetism. Springer International Publishing, 2021.

Metallic nanoparticles-decorated $Nd_xY_{1.00-x}Al_3(BO_3)_4$ sub-micrometric particles to enhance anti-Stokes excitation performance

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In the anti-Stokes excitation of trivalent rare-earth ions, the excitation photons energy is smaller than that of a given absorption transition, and the energy mismatch can becompensated by annihilation of phonons from the host lattice. Since the occupation number of phonons increases with the temperature, heating the system generally increases the efficiency of anti-Stokes excitation. Here, we exploited an intrinsic heating associated withlight-to-heat conversion in the interaction of a laser light with metallic nanoparticles (Ag or Au) on the surface of $Nd_xY_{1.00-x}Al_3(BO_3)_4$(x= 0.10, 0.20, and 1.00) sub-micrometric particles to enhance the anti-Stokes excitation efficiency with light at 1064 nm. Several upconversion emissions are observed from 600 nm to 880 nm, with the more intense at 750 nm due to the Nd^{3+}transition $\{^4F_{7/2}, ^4S_{3/2}\} \rightarrow {}^4I_{9/2}$. 300-fold enhancements are demonstrated for silver (Ag) nanoparticles, when compared with the pristine $Nd_xY_{1.00-x}Al_3(BO_3)_4$particles.

Acknowledgement:

Fundação de Amparo à Pesquisa do Estado de Alagoas (FAPEAL)
Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES)Fundação de Amparo a Pesquisa do Estado de Goias (FAPEG) Financiadora de Estudos e Projetos (FINEP)
Conselho Nacional de Desenvolvimento Cientıfico e Tecnologico (CNPq)

Mixed copper and platinum nanoparticles filmsanchored on silica substrate: synthesis and characterization

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The great interest of nanomaterials (NMs) is due to their different and modulated properties comparing to those of their bulk solids [1]. The class of metal nanoparticles (NPs) stands out because of their properties such as high chemical stability and high catalytic activity, as well as interesting optical properties resulting from surface plasmon resonance (SPR) [2,3]. Based on this, the present work aims to the synthesis and characterization of bimetallic and monometallic NPs of copper (Cu) and platinum (Pt) films, deposited on silica substrate. For the synthesis, silica coverslips were treated by piranha solution (2:1 H2SO4/H2O2), followed by the functionalization with 3-aminopropyltriethoxysilane (APTES) (10% v/v) simultaneously with the deposition of metallic precursors, in toluene solution and, finally, by the thermal reduction step at 300°C for a period of 15 minutes in a muffle furnace. Bimetallic films presented visually greater uniformity compared to monometallic Cu films, demonstrating the influence of Pt on the quality of synthesized films. The samples were characterized by UV-Vis spectroscopy, where the plasmon absorption signal was observed for all samples containing Cu. With the transmission electron microscopy (TEM) images, it was possible to observe the formation of a bimodal distribution of the NPs, whose crystallinity was observed by dark-field images, as well as the formation of organized 2D carbon structures, like graphene, resulting from the APTES pyrolysis. DRX, SEM and Raman measurements, in progress, will allow analyzing the crystallinity, homogeneity, composition and stability of the synthesized films.

Acknowledgements:
INCT Nanocarbono, INCT NanoVida, GQM-UFPR, CME-UFPR, LAMAQ - UTFPR.

References:

[1] ZARBIN, A. J. G., Química nova, v. 30, n. 6, p. 1469-1479 (2007)
[2] LI, L. et al., Journal of Electroanalytical Chemistry, v. 833, p. 393-400 (2019)
[3] CHAUDHURI, R. G. and PARIA, S., Chemical Reviews, v. 112, n. 4, p. 2373-2433 (2012)

Modulating white light emission temperature in $Ho^{3+}/Yb^{3+}/Tm^{3+}$ triply doped GeO_2-Nb_2O_5 materials for lighting applications

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Materials based on 0.3% Ho^{3+}/1.2% Yb^{3+}/0.5% Tm^{3+} triply doped with GeO_2–Nb_2O_5 were prepared by the sol gel methodology followed by thermal annealing at temperatures ranging from 700 to 1100 °C, which affected the structure and the white light generation of the solids. The spectroscopic properties of the samples depended on the final structure. In the photoluminescence spectra under ultraviolet excitation, it is possible to observe a characteristic broad band from 360 nm to approximately 600 nm, which overlapped some transitions of rare earth ions depending on the thermal treatment. This broad band was attributed to the emission of the GeO_2-Nb_2O_5 host lattice. In addition to the emission band of the host lattice, the emissions of the Ho^{3+} ions at 540, 590, 613, 623, and 653 nm were observed. The emissions of the Tm3+ ions at 653 and 704 nm transitions. The sample treated at 1100 ºC stood out: it generated white light through modulation of the warm white illumination under excitation at longer ultraviolet wavelengths (275 and 280 nm) and cold white under excitation at shorter ultraviolet wavelengths (245 to 270 nm). In this sense, changes in the excitation wavelength can generate the quality of white light that is necessary for a given lighting environment.[1]

Acknowledgements:
This work was supported by Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), and Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP). We also thank Companhia Brasileira de Metalurgia e Mineração (CBMM) for supplying Nb_2O_5 and Photonics Nacional Institute for Science and Technology (INFO).

Reference:
[1] SOUZA, V. S. et al. Journal of Luminescence, v. 248, p. 118978 (2022).

Nd^{3+} high order electrical susceptibility induced by CdS nanocrystals in a phosphate glass matrix

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In this work the nonlinear optical properties of a phosphate glass matrix doped with $Nd^{3+}$and codoped with CdS nanocrystals have been investigated by the time-resolved Z-scan technique[1]. Analyzing the transient signal, we show that it was possible to discriminate the thermal effect from the populational effects on the nonlinear refraction and absorption, allowing the determination of the parameters n_2', n_2'' and n_4'. We also investigated the dependence of these parameters on the relative concentration of Nd and CdS, and we observed a linear increase of these parameters with the CdS absorption. Evidence of energy transfer between Nd and CdS nanocrystals[2] was investigated by the determination of the non-radiative decay rate through lifetime measurements monitoring the emission at 1064 nm referring to the transition $^4F_{3/2}$□ $^4I_{11/2}$ of the Nd^{3+}. Acknowledgements:The authors are grateful to the CNPq, CAPES-Finance Code 001, FAPEMIG and INCT/Institute of Photonics (INFO) for the financial support.References:[1] Andrade, A. A.; Tenório, E.; Catunda, T.; Baesso, M. L.; Cassanho, A.; Jenssen, H. P. Discrimination between Electronic and Thermal Contributions to the Nonlinear Refractive Index of SrAlF5:Cr+3. J. Opt. Soc. Am. B, JOSAB 1999, 16 (3), 395-400.[2] Camilo, N. S.; Filho, J. C. S.; Lourenço, S. A.; Messias, D. N.; Pilla,V.; Silva, A. C. A.; Dantas, N. O.; Andrade, A. A. Study of the Optical and Structural Properties of the Phosphate Glass Doped with CdS Nanocrystals and Co-Doped with Nd3+Ions. Journal of Alloys and Compounds 2020, 158126.

NONLINEAR AND THERMO-OPTICAL PROPERTIES OF CARBON DOTS FROM BABASSU COCONUT (ORBIGNYA SPECIOSA) BIOMASS

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For this work, carbon dots were synthed by the hydrothermal method using biomass from the mesocarp of the Orbignya speciosa plant (babassu coconut) as a precursor, following a route proposed by Costa and colleagues [1]. The formation of the desired material was confirmed by UV-vis, FTIR, and Fluorescence, which indicated the formation of carbon dots, the presence of various functional groups on the surface, and their photoluminescence. The properties and stability of the material were characterized by TEM, DLS, and Zeta potential, which showed that the samples had an average diameter of approximately 63 nm and a Zeta potential of -34 mV. Recently, the optical nonlinearity properties of carbon dots have been highlighted, so Z-scan and thermal lens spectroscopy measurements were performed to extract the nonlinear optical (n2) and thermo-optical parameters. The carbon dots showed a nonlinear response ranging from -5.6×10-12 cm2/W to -6.4×10-12, obtained by fitting the experimental data using the standard model proposed by Cuppo and colleagues [2]. The thermal diffusivity values obtained were approximately 1.4×10-3 cm2/s, indicating the potential of the material for photonics applications and thermal insulation.

References:

[1] Costa, R. S., de Castro, M. O., da Silva, G. H., Delite, F. D. S., Strauss, M., Ferreira, O. P., ... Viana, B. C. Carbon Trends, 5, 100133 (2021).

[2] Cuppo, F. L. S. A., Neto, A. M. F., Gómez, S. L., Palffy-Muhoray, P. JOSA B, 19(6), 1342-1348 (2002).

Acknowledgments:
UFPI, CNPq, CAPES, FAPEPI, FISMAT

Nonlinear optical characterization of a new organic crystal with non-centrosymmetric structure

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This work presents the optical properties of a simple, but interesting organic salt. The crystals were built up from 2-aminopyrazine as a cation, with N1 protonation and dihydrogen phosphate as anion [1]. The crystals exhibit a second-order optical nonlinearity and could therefore be useful for photonic applications. This response occurs due to the dipole formed by the interaction of aminopyrazine and phosphoric acid, a system resembling the renowned KDP crystal. As a consequence, it was possible to measure a non-centrosymmetric phase showing second harmonic generation in powder formed from microcrystalites of the new material. The nonlinear optical characterization was performed in two experiments. Firstly, the powder was exposed to optical trains of Q-switched mode-locked pulses of varying intensity from a Nd:YAG infrared laser (1064 nm, 900 mW). The SHG light was detected by a photomultiplier in reflection and in transmission through the sample. The FWHM of the green pulse train was ~1.33 shorter than that of the IR pulses, a value close to the theoretical value ($2^{0.5}$) expected for frequency doubling. Also, the polarization of the incident light was changed 360° in steps of 5° and the SH intensity was monitored. A large angular dependence of the SHG light intensity on this parameter was observed, with the ratio Imax/Imin approaching ~9 for one polarization angle.

Acknowledgments:
Alexandre Camara thanks to Fundação Carlos Chagas de Amparo à Pesquisa do Estado doRio de Janeiro (SEI- 260003/015620/2021), CAPES - Finace Code 001. The authors thanksCNPq and FAPEG for financial support.

References:
[1] A. K. S. M. Valdo, A. M. Sarotti, F. T. Martins, CrystEngComm, 2017, 19, 5960-5965. DOI: 10.1039/c7ce01490b.

Obtaining and characterization of nanostructured powders of CuNb2O6by proteic sol-gel synthesis

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Niobium has been considered a promising material for science due to the good results indicated in current studies. For Brazil, expanding knowledge and applicability of Niobium (Nb) is of great importance since the country is the largest holder of the ore in the world, around 98%. Copper niobate ($CuNb_2O_6$), in turn, is one of those Nb compounds that are gaining prominence in science because its properties enable its application in technological sectors. The process of obtaining $CuNb_2O_6$can result in different phases and properties, so in this study, therefore, nanoparticles of $CuNb_2O_6$were produced using the proteic Sol-Gel synthesis route modified with gelatin from the precursors Niobium Pentoxide and Oxide of copper. After synthesis, the powders were calcined in a muffle furnace at temperatures of 800 °C, 900 °C and 1000 °C, for 3 h and a heating rate of 5 °C/min. Subsequently, they were characterized using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS) and Raman Spectroscopy. XRD results show that, at 800 °C, the monoclinic phase of CuNb2O6 is identified, however, this was only completely obtainedwhen calcined at 900 °C. The samples calcined at 1000 °C resulted in the complete orthorhombic phase. All samples with total formation of polymorphs showed good dispersivity, with no evidence of contamination, the morphologies of the characteristic particles and the Raman spectra were consistent with the literature.

Acknowledgements:
UFRN; PPGCEM; CAPES; LMCME.

References:
[1] NASCIMENTO, E. S. DO et al. Brazilian Journal of Development, v. 7, no. 11, p. 109468-109476(2021).
[2] KAMIMURA, S. et al. Journal of Photochemistry and Photobiology A: Chemistry, v. 356, p. 263-271(2018).
[3] NWANNA, EMEKA CHARLE IMOISILI, PATRICK EHI; BITIRE, SARAH; JEN, TIEN-CHIEN. Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, v. 44, n. 2, p. 3610-3627(2022).

Optical characterization of NaGdF4-based core-shell nanoparticles with energy upconversion properties

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Inorganic nanocrystals (NCs) of various compositions have become an increasingly studied research topic in recent years with special attention given to lanthanide compounds. Their luminescence properties can be tailored for several applications like solar cells, photocatalysis, in vitro and in vivo biological markers, etc. Accurate determination of their internal structure and chemical composition is fundamental to the understanding and development of new nanomaterials. It has been demonstrated that the growth of a shell surrounding the core nanoparticle is a common way to improve the luminescence efficiency of such materials. This design introduces the possibility of constructing novel upconversion nanoparticles. The luminescent mechanisms of lanthanide ions can be divided into two categories: energy upconversion (UC) and energy downconversion (DC). Lanthanide upconversion luminescence is an anti-Stokes shift process where lanthanide ions absorb lower energy photons and emit higher energy photons. In this study, core and core-shell-structured lanthanide-based nanocrystals with energy upconversion properties were synthed by traditional high-temperature thermolysis using oleic acid, octadecene, and lanthanide salts as precursors. The NaGdF4 matrix was used to form NaGdF4:Yb3+,Er3+ core-only, and NaGdF4:Yb3+,Er3+@NaGdF4 core-shell nanoparticles with hexagonal (β phase) crystal structure as confirmed by XRD diffraction. Optical properties were studied by UV-Vis and photoluminescence spectroscopy. The upconversion mechanism is due to energy transfer from Yb3+ to Er3+ while the passivation of surface defects on the core by the shell increases the luminescence efficiency. In these materials, green (540 nm, 4S3/2 → 4I15/2 transition) and red (650 nm, 4F9/2 → 4I15/2 transition) emissions were detected upon excitation at 976 nm (2F7/2 → 2F5/2 transition of Yb3+).

Optical properties of silver indium sulfide quantumdots coated with cadmium chalcogenides

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I-III-VI ternary quantum dots have optical and electronic properties modulated through chemical composition and scale. The production of heterojunctions improves the optical properties of nanosystems, being the formation of core/shell systems the most suitable for nanocrystals of this type. [1] In this work we report the modulation of optical properties of AIn_5S_8(AIS) nanocrystals when coated with CdX seeds (X: S^{2-}, Se^{2-}). The synthesis were performed in electrochemical cavity cell, and stabilized by L-glutathione. The formation of the AIS/CdX heterojunction was carried out so that the core/shell ratio, with respect to the chalcogenides, was 1:0.25, 1:0.5, 1:1, or 1:1.5. After the formation of AIS/CdX, a hypsochromic shift was observed in the emission spectra for the AIS/CdS systems ($\lambda_{em}$from 585 nm to 571 nm) and bathochromic for the AIS/CdSe systems (λem from 585 nm to 560 nm). The AIS QDs obtained quantum yield (QY%, R6G pattern) equal to 4.06%. After CdS and CdSe coatings, the QY ranged from 9.36% - 11.6% and 2.70% - 0.78%, respectively. X-ray diffractograms presented the cubic structure formation of AgIn5S8, and after the coverage was verified the displacement of the diffraction peaks to lower 2θ values when coated with CdS, and to high 2θ values when coated with CdSe. The structural evolution is associated with the difference between the ionic radii of $Se^{2-}$and S^{2-}. In the time-resolved photoluminescence experiments, AIS showed a strong dependence on electronic defects, with 97.01% of relative abundance its associated with donor and acceptor states (DAP). The average lifetime increases when in the presence of coatings, due to the increase in lifetimes associated with processes mediated by defects. The presented properties demonstrate the potential of coatings in modulating the optical properties of semiconductor nanocrystals, which can be enhanced as in this work in the presence of CdS.

[1] J. Alloys Compounds, 853, 2021, 156926

Optical spectroscopy applied in curcumin with metallic ions characterization

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Curcumin has, recently, drawn attention and attracted research investments around the world due to it abundant properties in both optical and biological science. Some of its biological applications in traditional and modern medicine involves antitumor, antioxidant, antiviral and anti-inflammatory properties and can even be used in cases of Alzheimer’s, psoriasis and diseases of the digestive system. The effects of curcumin are directly connected with its form of administration, however, its low solubility in water results in low bioavailability, as it is poorly absorbed by the body in the most common forms available. A possible solution is the production of stable complexes formed by curcumin and metals such as magnesium (Mn), iron (Fe), copper(Co), zinc (Zn) among others from groups 7-12 of the periodic table. In addition, to increase its bioavailability, these complexes allow diverse biotechnological applications from medicines to sensors. The materials chosen for this study are complexes constituted by curcumin and a metal and, for the optical characterization of the complexes, we used optical techniques such as photoluminescence, ellipsometry, Raman. Chemical techniques were used for a better structural understanding, such as magnetic resonance. Some optical characterization exploring a possible magnetic circular dichroism and other effects involving magnetic field, were also be performed. The low number of studies that explore this type of complex, in terms of the radiation interaction with matter, turn this study essential to understand its mechanisms and may shed new light onthe exploration of curcumin, within the applications mentioned in the text

Optical thermometry of tellurite glasses doped withEr^{3+}-Yb^{3+} under UV and NIR excitation

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The use of glasses doped with rare earth ions (REI) as optical sensors have a high potential to be implemented as optical fibers. Tellurite glasses offer excellent optical, thermal, and structural properties in this scenario. The ability to incorporate large amounts of REI is a notable candidate for these purposes. One of the most used techniques for optical thermometry is the fluorescence intensity ratio (FIR). The main advantage of using this technique is that it is not affected by external factors such as energy fluctuations of the excitation sources [1]. In this research, tellurite glasses doped with Er^{3+}-Yb^{3+}were fabricated to study their thermal sensing performance under 380 and 980 nm excitation. The FIR was applied on the 2H11/2 and 4S3/2 thermally coupled levels of the Er3+ in the temperature range of 93 - 348 K. These glasses exhibit high thermal sensitivities, and their values depend on the REI concentration and excitation source. Furthermore, the refractive indices (>2.04 at 532 nm) and thermal stability are higher compared with other Er3+-Yb3+ doped tellurite glasses for thermal sensing applications. These features become our glasses propitious candidates for optical fiber, luminescent material, and thermal sensing.

Acknowledgments:

This research was supported by the São Paulo Research Foundation (FAPESP) under the grants 2013/07276-1, 2020/04835-3 and 2022/05478-5.

References:

[1] C. Wang et al., J. Alloys Compd. 894, 162494 (2022).

Precipitation of pure metal on glass by ns-laser irradiation

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Laser processing of glasses have been widely used to promote local changes on material properties and structure, promoting applications from photonic devices to microfluidics. Advances on laser technology have expand the glass processing possibilities, enabling to achieve 3D manufacturing, bulk or surface crystallization. Moreover, laser-induced crystallization is relevant to functionalize glass matrices through the precipitation of different micro/nanocrystals, such as $LiNbO_3$and β- $BaB_2O_4$with second harmonic generation [1]. Concerning the nonlinear optical properties, it is also important to search for materials with significant third-order susceptibility, in which heavy-metal oxide glasses have excelled due to their hyperpolarizabilities. Previous results on 50-BO1.5-50PbO glass showed that fs-laser associated with chemical etching in KOH solution resulted in the architecture of lead oxide microcrystals [2]. In order to determine the influence of pulse duration on the crystallization process, we used a ns-laser to irradiate the 50-BO1.5-50PbO glass. Laser pulses of 10 ns at 1064 nm, with repetition rate of 10 Hz were focused on the glass surface, which was translated regarding the beam at constant scan speeds of 0.2-7 mm/s. Results shown that independent of scan speed, the bottom surface became crystalline, which has been discussed to be due to self-focusing effect associated with the high nonlinear refractive index of the glass (n_2= 4.7 x $10^{-19}m^2/W$ at this wavelength). The formation of metallic lead (Pb) was identified by x-ray diffraction, being responsible for changes the crystallization process in KOH solution previously investigated. The authors acknowledge the support from FAPESP 2018/11283-7, CAPES and CNPq. [1] E. Muzi et al. OptMatExp, 11, 1313, 2021.

[2] J. M. P. Almeida et al. Architecture of lead oxide microcrystals in glass: a laser and etching based method. CrystEngComm, 18, 5959, 2016.

Preparation of AgInTe2 quantum dots in an aqueous medium

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Quantum dots (QDs) are semiconductor nanocrystals with sizes ranging from 2 to 10 nm, which have charge carriers (electrons and holes) in a quantum confinement regime, giving them optical properties, that can be modified just by varying their size or chemical composition. Furthermore, QDs exhibit high photostability, having resistance to photobleaching over a period of minutes to several hours, and have a chemically active surface that can be conjugated to molecules of interest, increasing their affinity with the analyte. Due to their properties, QDs have been used for a wide range of applications, like sensors, cellular markers, and solar cells. [1,2]. On the other hand, the most established QDs in the literature are prepared in organic solvents and with harsh conditions, and/or have high toxicity due to the presence of heavy metals in their constitution, such as lead and cadmium, which lower their potential for the majority of applications. In this context, ternary QDs like silver and indium chalcogenides, appear as a solution, since they possess excellent fluorescence and very low toxicity. Thus, this work had the aim of developing a synthetic procedure to prepare AgInTe2 in an aqueous medium. For this, a chemometric study was carried out, varying temperature, pH, synthesis time, and precursor concentration, in order to optimize the optical properties of the AgInTe2 QDs. From the optical characterizations, promising results were found in relation to the fluorescence intensity, allowing the produced material to be used, among other applications, as an optical sensor.

Acknowledgments:

The authors are grateful to CAPES, CNPq - GD, FACEPE, UFPE, and CESAM/FCT/MCTES (UIDP/50017/2020+UIDB/50017/2020+LA/P/0094/2020) for financial support.

References:
[1] Pereira, M. G. C. et al. Elsevier, 2016 v. 1, p. 131-158
[2] Tong, Xin and Wang, M. Zhiming. Springer, 2020, 310, 1-5.

Properties investigation of strontium borotellurite glasses

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This work aims to study the synthesis and structural and optical properties of a vitreous system formed by B_2O_3, TeO_2 and SrO. The samples were synthesized by melt-quenching method with the following composition (X)SrO-(Y)B_2O_3-(Z)TeO_2 where X=10, 16, 26 and 30; Y=20, 29, 44 and 50; Z=70, 55, 30 and 20wt%, respectively. The glass samples were characterized by X-ray diffraction, Raman spectroscopy, optical absorption spectroscopy, volumetric density, and refractive index. A ternary phase diagram of the synthesized samples was elaborated. The results obtained were discussed from the concentration of TeO_2 of the glasses. From the analysis of X-ray diffraction it was possible to confirm the amorphous character of the samples [1]. Raman spectra present bands associated to BO_3, BO_4, TeO_3, TeO_{3+1} and TeO_4 units. The optical absorption spectra have a wide optical transmission window in the range of ~590 nm to 2050 nm. Refractive index and volumetric density values increase linearly as a function of the increase of the TeO_2 concentration, which occurs due to substitution of B_2O_3 and SrO by TeO_2. The results suggest that these glasses are excellent candidates for rare-earth doping and optical applications [2].

Acknowledgements: We are thankful to the Brazilian agencies CNPQ, FINEP and FAPEMA for financial support of this study.

References:

[1] K. O' Connell, M. Hanson, H. O' Shea, D. Boyd, Linear release of strontium ions from high borate glasses via lanthanide/alkali substitutions, J. Non.Cryst. Solids. (2015). https://doi.org/10.1016/j.jnoncrysol.2015.09.017.

[2] E.C. Paz, T.A. Lodi, B.R.A. Gomes, G.H.A. Melo, F. Pedrochi, A.Steimacher, Optical and spectroscopic investigation on Calcium Borotellurite glass system, Solid State Sci. (2016). https://doi.org/10.1016/j.solidstatesciences.2016.02.014.

Size versus Surface Defects Balance of CdSe Quantum Dots for Photocatalytic Organic Reactions

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Sulfones are a group of compounds of pharmacological interest and are produced by Friedel-Crafts sulfonylation of arenes. Photocatalysts, as quantum dots (QDs) gain relevance for their efficiency and degree of recovery. This work presents the combined effect of the chemistry of surface defects on cadmium selenide QDs (CdSe QDs) and their impact on photocatalyst performance for sulfonylation reactions. Cysteamine-stabilized CdSe QDs were electrosynthed and the combined effect of surface defect and chemistry was controlled for time of heat treatment under reflux (t = 0.5, 1, 2, and 4 h). The reaction between benzyl bromide and sodium p-toluenesulfinate, photocatalyzed (365nm, 6W) by CdSe QDs, was used as a reaction model.[1] CdSe QDs showed a progressive decrease in optical bandgap from 2.45 eV (0.5 h) to 2.30 eV (4 h). At the same time, the combined effect of increasing the full width at half maximum (FWHM) of the emission band and the emission wavelength (λem) from 120nm/631nm (0.5 h) to 126nm/650nm (4 h) was verified, which are associatedto the rise of surface defects, particle growth and Stokes shift. Thus, the heating time leadsto a decrease in the quantum yield, in addition to the increase of the luminescence half-lifetime. The light-off sulfonylation reaction in presence of CdSe QDs led to 12% yield of benzyl-p-toluenesulfone (2 hs reaction time). Under UV light (365 nm), the photocatalysis yields decreased from 99% to 92%, according to the increasing of CdSe nanoparticle and decrease of quantum yield. CdSe intragap recombinations diminish the transfer of electrons on the QD surface, thus decreasing the efficiency of the photocatalytic reactions, which is a proof-of-concept of the influence of the surface defects on sulfonylation reactions photocatalyzedby QDs.

Acknowledgments:
MCTI, MEC, CNPq.

References:

[1] Catal. Sci. Technol., 2023, 7, 1-8. 10.1039/d3cy00059a

Spatial self-phase modulation of AuNPs obtained by photochemical synthesis with different wavelengths

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The interesting physical and chemical properties displayed by nanomaterials have provided an increase in their applications in various fields of science and hence an increase in the search of synthesis routes favoring a greater control of size and shape of the nanoparticles. Among the various available routes of synthesis, the photoinduction has been disclosed as simple and powerful technique, allowing to obtain monodisperse nanoparticles. Spatial self-phase modulation (SSPM) is a phenomenon characterized by the appearance of diffraction rings in the far field when a Gaussian profile laser focuses on a non-linear medium. By relating the number of rings formed with respect to a given power of the excitation laser, it is possible to determine the module of the so-called non-linear refractive index n2 of the sample. The sign of n2 can be determined from the laser-induced thermal lens. The modification of the laser wavefront causes it to undergo convergence or divergence, depending on whether the refractive index of the medium is positive or negative, respectively, which is related to n2. The emergence of rings is due to the phase variation $\Delta\varphi NL$ acquired when passing through the medium. The technique is very sensitive with dilute samples, which makes it an excellent spectroscopic tool for characterizing liquid samples. Although SSPM has been developed as a typical method for nonlinear optical response, the origin of the change in refractive index due to the phenomenon is still little explored. In this work we present the photothermal characterization through the spatial selfphase modulation technique of gold nanoparticles (AuNPs) obtained through the photochemical reduction of the precursors dispersed in water. Particle size and shape influence the optical response of the fluid, facilitating or hindering the appearance of diffraction rings due to laser excitation.

Spectroscopic properties investigation of MnO doped alkaline borate glass

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Glasses doped with transition metal (TM) ions have been widely investigated because of their interesting spectroscopic and optical properties, which make them favorable candidates for application in the fields of opto-electrical and electrochemical devices. Among TM ions, manganese is attractive since it exhibits different valance states depending on the properties of the glass matrices [1,2]. In this work, an alkaline borate glass was doped with different concentrations of MnO (0.05, 0.10, 0.25 and 0.50 %mol) to evaluate the spectroscopic properties as a function of the dopant concentration. The samples were prepared by the traditional melt-quenching method in air atmosphere and their properties were investigated by DRX, FTIR, optical absorption, excitation and luminescence spectra, and CIE chromaticity diagram. The absorption spectra present characteristic transitions of $Mn^{2+}$and Mn^{3+}ions, and the bands in the luminescence spectra change as a function of the excitation source. The results are promising for the material application on technological devices.

Acknowledgements:
This study was partly financed by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001. We are thankful to the Brazilian agencies CNPq, CAPES, FINEP and FAPEMA for the financial support.

References:

[1] M.R. Ahmed, B. Ashok, S.K. Ahmmad, A. Hameed, M.N. Chary, M. Shareefuddin, Infrared and Raman spectroscopic studies of Mn2+ ions doped in strontium alumino borateglasses: Describes the role of Al2O3, Spectrochim. Acta - Part A Mol. Biomol. Spectrosc. 210(2019) 308-314. https://doi.org/10.1016/j.saa.2018.11.053.

[2] C. Parthasaradhi Reddy, V. Naresh, K.T. Ramakrishna Reddy, Li2O-LiF-ZnF2-B2O3-P2O5: MnO glasses - Thermal, structural, optical and luminescence characteristics, Opt. Mater. (Amst). 51 (2016) 154-161. https://doi.org/10.1016/j.optmat.2015.11.035.

STRUCTURAL AND PHOTOLUMINESCENCE PROPERTIES OF Tm_2O_3-DOPED IN OXYFLUORIDESYSTEM (P_2O_5-KF-Al_2O_3)

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Materials science has experienced important advances in recent years, notably, many works are directed for materials with applications aimed at visible light sources [1]. The Tm^{3+}can have a strong blue emission, which allows the development of lasers and LEDs in this color region which makes it possible to use this element in glass matrices aimed at photonic applications [2]. This study refers to the synthesis of a vitreous series in oxyfluorides phosphate system, (38)P_2O_5-50-xKF-12Al_2O_3-xTm_2O_3(PKAl:Tm), with x= 0.25, 0.50, 0.75 and 1 mol%, by the melt-quenching method. The synthesized materials showed amorphous characteristic verified by XRD data. The absorption spectra show six main bands characteristic of Tm^{3+}ions, from ground state $^3H_6$to excited levels $^3F_4$, $^3H_4$, $^3H_5$, $^3F_{3,2}$, $^1G_4$and $^1D_2$, which shows an intensity increase with the concentration. In the FTIR spectra, it was verified that the profiles of the PKAl samples are similar to glasses based on phosphate systems. The excitation spectra of PKAl:Tm glasses show an intense band centered at 358 nm ($^3H_6 \rightarrow {}^1D_2$). The luminescence spectra with excitation of 358 nm show three bands at 453 nm ($^1D_2 \rightarrow {}^3F_4$), 480 nm ($^1G_4 \rightarrow {}^3H_6$) and 660 nm ($^4F_{9/2} \rightarrow {}^6H_{11/2}$); the highest emission intensity was observed to the sample doped with 0.75 mol% of Te2O3. The calculated CIE diagram shows that the doped samples have coordinates close to the primary blue color, which favors the application for the development of blue LEDs and wLEDs. These results show that the PKAl:Tm glasses are a good candidate for optical and photonic applications.

Acknowledgements:CAPES, CNPQ, FAPEMA, PPGCM-UFMA and LEOF.

References:

[1] Z. Xiao, J. Liu, S. Qin, X. Xu, H. Chang, J. Liu, T. Liu, L. Han, Q. Zhang. J. Non. Cryst. Solids. 597 (2022) 1-9. doi:10.1016/j.jnoncrysol.2022.121920. [2] R. Praveena, K.H. Jang, C.K. Jayasankar, H.J. Seo. J. Alloys Compd. 496 (2010) 335-340. doi:10.1016/j.jallcom.2010.02.007.

Study of nanocomposites based on gold nanoparticles, europium complex, and polypyrrole for optical sensors applications

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Hybrid nanocomposites formed by a polymeric matrix and metallic nanoparticles are a class of materials that have received great interest for optical sensors, electronic devices, and biomarker nanosensors. In this study, we report the synthesis and characterization of luminescent nanocomposites based on gold nanoparticles (AuNPs) anchored or adsorbed to the luminescent complex $[Eu(tta)_3(H_2O)_2]$, where tta = thenoyltrifluoracetone, in the presence of the polymer polypyrrole (Ppy), seeking synergism between the phases and improvement of properties compared to isolated chemical components, for sensing applications and to perform electronic devices based on optical fiber. The nanocomposites were synthed by varying the amount of monomer pyrrole, gold precursor $HAuCl_4$and europium complex. The AuNPs plasmon band was identified by UV-Vis spectroscopy, and absorption bands from the tta ligand and Ppy polymer also confirm hybrid formation. The AuNPs were identified by TEM results, where crystalline structures were observed in dark field images for all samples. Moreover, this technique provided evidence of the polymer acting as a stabilizer. Photoluminescence spectra displayed intra-configurational transitions of the Eu^{3+} ion observed for all sample conditions, except for those where a higher amount of pyrrole was added in the synthesis (36 μL of pyrrole 5.0×10^{-2}mol L^{-1}), explained as a result of quenching mechanisms. Confocal optical microscopy images from hybrid nanocomposite evidence europium complex presence. Furthermore, AFM images suggest that the presence of the complex promotes changes to the sample's surface, making it rougher and thicker.

Acknowledgments:
CAPES, CNPq, LAMAQ, DAQBi-UTFPR, QI-UNESP, CME-UFPR.

References:
[1] ATES, B. et al, Chemical Reviews, 120.17, 9304-9362 (2020).
[2] INAGAKI, C. S., OLIVEIRA, M. M.; ZARBIN, A. J. G, Journal of colloid and interface science, 516, 498-510 (2018).

Study of silver nanoparticles synthesis through photochemical method

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The study of metal nanoparticles is of great interest both scientifically and technologically due to their unique properties related to Localized Surface Plasmon. Therefore, precise control over the shape and size of nanoparticles is necessary, which requires special attention during synthesis. In this study, the synthesis of silver nanoparticles was investigated through the photochemical method, in which photons are absorbed by a photochemically active compound, inducing the reduction of metal ions. To obtain silver nanoparticles, a solution of 1 mMol of $AgNO3$, sodium citrate and PVP were used, and the solution was exposed to the light of a LED. Some parameters were changed in different syntheses, such as the wavelength of the LED, the quantity of sodium citrate and PVP, and the exposure time to the LED light. After synthesis, UV-visible spectrometry was performed to observe the formation of nanoparticles, dynamic light scattering (DLS) to observe the size of nanoparticles, and Zeta potential measurement to verify the stability of nanoparticles. It was observed that increasing the quantity of sodium citrate led to a decrease in the absorption peak in UV-visible spectrometry and an increase in the mean size of nanoparticles. The same effect was observed with an increase in PVP. With variation in the wavelengths of the LEDs, the samples made with the UV LED showed a much higher absorption peak in UV-Vis spectrometry than the samples made with other wavelengths.

STUDY OF THE SYNTHESIS AND CHARACTERIZATION OF COPPER NIOBATE VIA THE SOL-GEL METHOD FROM THE PREPARATION OF THE NIOBIUM PRECURSOR

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This work aims to study the synthesis of Copper Niobate (CuNb2O6) via the proteic Sol-Gel method, which is a wet chemical route little used for the preparation of this material, but stands out for being a green synthesis, starting from the preparation of the Niobium Ammonium Oxalate precursor. Niobate has stood out in science due to its various properties and technological applications, such as in solar cell devices, microwaves and infrared lasers. Therefore, the need to produce this nanomaterial with a simpler and low-cost methodology became greater. For this, Niobium Ammonium Oxalate precursor powders were used, prepared from melting and complexation processes, and commercial Copper Nitrate, both of which have high solubility in water, in addition to the addition of agar-agar as a chelating and polymerizing agent. . After obtaining the gels, the powders received the appropriate thermal treatment, were calcined in a muffle at temperatures of 700 °C, 800 °C, 900 °C and1000 °C, for 3 hours at a heating rate of 5 °C /min. Subsequently, the obtained powders were characterized by RAMAN Spectroscopy, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive Chemical Microanalysis (EDS). so it was found that for the calcined niobate at 900 °C complete formation of the product in the monoclinic phase occurred, and at 1000 °C CuNb2O6 formed completely in the orthorhombic phase. These samples with complete formation of the compound showed good results, such as good dispersion, high purity, no evidence of contamination and characteristic morphologies, consistent with the literature.

Substrate type influence on indium oxide films,obtained from resistively evaporated indium.

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Indium oxide (In2O3) in stoichiometric form is an insulator, whereas in the non-stoichiometric form it behaves as a wide bandgap semiconductor (>3eV), with high transparency in the visible and high reflectivity in the infrared. These properties lead to an expressive applicability [1]. Nanomaterials based on In2O3 have received considerable attention for technological applications [2], such as thin film transistors (TFT) [3], transparent conductors and ultraviolet sensors, among others.The main parameters considered are the substrate type for film deposition: silica or soda-lime glass, along with the thickness of the In film, deposited by resistive evaporation. To obtain In2O3, In filmswere kept at a temperature of 300°C for 2.5h (under room atmosphere conditions), which isreached at a rate of 1°C/min starting from room temperature. Measurements of X-ray diffraction (XRD) allow verifying that films 200nm thick present characteristic planes of twostructures: from the originally deposited metallic Indium and from In2O3, forming a nanocomposite of both structures. For 50nm and 100nm thick films, XRD data reveals onlythe formation of In2O3, differing in the intensity of the existing peaks. For more uniformoxidation, diffusion throughout the film should be made possible, where the oxygen atoms would need larger energy, or more time. Films deposited on soda-lime glass have bettercrystallization than films deposited on silica. One of the main causes of this observation maybe related to the difference in roughness of the used substrates. Soda-lime glass has an average roughness higher than silica. The increase in roughness directly affects the increase in surface area, increasing the availability for oxygen atoms to adsorb on the surface, originating the oxidation of the In layer to In2O3.References[1] G. Kiriakidis et al.,Mater. Phys. Mech. 1, 83-97(2000)[2] C.-H. Ho et al., J. Phys. Chem. C 115, 25088-25096(2011)[3] G. Shen et a., ACS Nano 5, 6148-6155 (2011)

Sustainable urethanesil based on castor oil doped with europium complex and upconverting microparticles for photonic applications

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Polymers based on vegetable oils can be excellent suitable candidates for replacing fossil polymers, for example, polyurethanes (PU) based on castor oil (CO). However, the synthesis of PU materials is normally carried out using organic solvents, metals and high temperature[1]. To overcome these problems, the sol-gel route is an alternative approach based on mild conditions to produce environmentally friendly organic-inorganic hybrid urethanesil (Ut) [1]. Therefore, in this study, we proposed the synthesis of a sustainable organic-inorganic hybrid (OIH) based on Ut derived from CO, as well as the incorporation and interaction of europium β-diketone $[Eu(tta)_3(H_2O)_2]$ and UCMPs NaF_4 : Yb/Er[3]. Ut hybrid was synthesized from the reaction of 3-(triethoxysilyl)propyl isocyanate (ICPTES) and CO in a 3:1 molar ratio. The films were obtained by the sol-gel process, following the casting method[1]. The CO-based OIH films showed high transmittance in the visible and infrared (90%), without major changes with the insertion of dopants and the Ut matrix. The results also revealed an increase in the intrinsic quantum yield of PL (Q^{Eu}_{L}) for $[Eu(tta)_3(H_2O)_2]$. Is has been verified a value of 27% for the isolated complex, while when it is incorporated into OIH PU a value of 49% was observed[2]. In addition, the UCMPs of 8 um was successfully doped, as observed by UC luminescence results.

SYNTHESIS AND CHARACTERIZATION NON-LINEAR OPTICAL OF BIMETALLIC SILVER AND GOLD NANOPARTICLES BIOSSYNTHESIZED FROM HYMENAEA COURBARIL (JATOBÁ)

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Green synthesis has been considered an innovative and sustainable alternative for producing metallic nanoparticles (NPMs) due to the use of phytochemical compounds or their parts as reducing agents and stabilizers [1, 2, 3]. This study reports, for the first time, the use of an aqueous extract of Hymenaea courbaril (Jatobá) as a reducing agent and stabilizer in the synthesis of metallic gold (AuNPs), silver (AgNPs), and bimetallic (Ag@AuNPs) nanoparticles, and investigates their non-linear optical properties with continuous laser excitation. The presence of Localized Surface Plasmon Resonance (LSPR) bands confirmed the formation of metallic nanoparticles that are characteristic of these nanomaterials. DLS measurements revealed varied s and polydispersity of the nanoparticles, and all nanostructures have a negative surface charge. TEM images showed that the nanoparticles have a spherical shape. Thermo-optical properties were investigated using thermal spectroscopy techniques, which showed thermal diffusivity values of the order between (8-10) x10-4 cm²/s. The Z-scan Technique demonstrated that the non-linear refractive index is negative. The non-linear effects of the nanostructures indicate that they can be controlled depending on the composition of the nanostructure. The results indicate that nanoparticles can be applied in systems for optical limitation, studies in non-linear optics, and Photothermal Therapy.

Agradecimetos: FAPEMA, PPGCM, FISMAT, LBM, LIMAV, UFPI e UFPR.

[1] ASHIKBAYEVA, Z. et al. Optics and Laser Technology, Volume 161 (2023).
[2] NAILA, S. et al. Applied Organometallic Chemistry, Volume 37 (2023).
[3] SUNDARRAJAN, S. K. ; POTTAIL, L. Applied Nanoscience, Volume 11 (2021).

SYNTHESIS AND CHARACTERIZATION OF BENZOTHIOPHENE-BENZOTHIADIAZOLE CONJUGATED POLYMERS FOR POTENTIAL APPLICATION IN PHOTOVOLTAIC DEVICES

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The growing need for energy generation using renewable resources has motivated research at a global level. Energy conversion using non-renewable sources (fossil fuels) has contributed, on a large scale, to air emissions and other environmental risks over the years. One of the alternatives for generating clean energy is organic solar cells (OSCs), formed by an active layer composed of a donor material alongside an acceptor material, prepared in thin films on a nanometer scale. These devices feature advantages such as flexibility, low cost, and reduced weight, among other benefits. Conjugated polymers used in bulk heterojunction (BHJ) solar cells, based on units containing fused benzodithiophene and benzothiadiazole rings, stand out due to great results achieved in energy conversion efficiency (PCE), as presented in the literature [1, 2]. In this work, copolymers containing benzodithiophene and benzothiadiazole units were synthed, and the following synthesis parameters were evaluated: volume of solvent, mass percentage of catalyst used, and two types of catalysts. Polymerization reactions were carried out according to the Stille coupling. The obtained polymers were purified by Soxhlet extraction with methanol, acetone, hexane, chloroform, and chlorobenzene. The conjugated polymer fractions in chloroform were characterized by 1H-nuclear magnetic resonance (1H-NMR), thermogravimetric analysis (TGA), ultraviolet spectroscopy (Uv-vis), and cyclic voltammetry. The polymer yields were near 100%, although an insoluble fraction was obtained in the reaction with a low amount of solvent in the polymerization. The other reaction conditions produced only chloroform-extracted polymer, which is the one mostly used to manufacture OSC devices.

[1] Q. Liu, Y. Jiang et. al, Science Bulletin vol. 5, 272 (2020)
[2] X. Zhong, T.W. Chen et al, ACS Applied Polymer Materials vol. 65, 1937 (2023)

Synthesis of Niobium Phosphate Glasses and Structural Characterization by Solid State Nuclear Magnetic Resonance

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Phosphate glasses are widely used in optical communications and solid-state lasers owing to the high solubility of photonically active metal ions in them [1]. The incorporation of transition metal ions, such as Nb_2O_5, not only improves the chemical durability of phosphate glasses, but also significantly contributes to increase both their linear and non-linear indexes of refraction [2]. However, a fundamental understanding of the structural role of niobium, which can serve either as a network modifier or as a network former, in these glasses has not been achieved. Solid-state nuclear magnetic resonance (NMR) has been proven to be a powerful tool for structural elucidation of glasses, due to its element-selectivity, inherently quantitative character, and its focus on local order [1]. From the NMR point of view, niobium features one of the most NMR-sensitive nuclei (^{93}Nb) which is 100% natural abundant. Nevertheless, it suffers from a large nuclear quadrupolar moment, and the ^{93}Nb NMR spectra are dominated by strong quadrupolar interactions, resulting in excessive line broadening and poor resolution. These challenges can be addressed by techniques involving fast MAS, wideband excitation methods and dipolar recoupling techniques [3]. Here, we report results on glasses in the system Na_2O-Nb_2O_5-P_2O_5 from two compositional series. Advanced NMR experiments have been used to characterize the local environments of sodium, phosphorus and niobium with the aim of obtaining new structural insight towards the development of new structure-function correlations.

Acknowledgements:

This work is supported by the São Paulo Research Foundation (FAPESP) under grants numbers 2013/07793-6 (CEPID program) and 2022/01937-5

References:

[1] Zhang R and Ren J, The Journal of Physical Chemistry, v. 121, p. 10087-10094 (2017)

[2] Koudelka L and Kalenda P, Journal of Non-Crystalline Solids, v. 459, p. 68-74. (2017).

[3] Koppe J and Hansen MR, The Journal of Physical Chemistry A, v. 124, p. 4314-4321(2020).

Thermal diffusivity of silver nanofluids by thermal lens spectroscopy

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The thermal properties of a base fluid can be enhanced by using nanoparticles. These properties can be tuned, for example, by the concentration, and shape of the nanoparticles [1]. In the case of thermal diffusivity, it can be obtained by measuring independently the thermal conductivity, heat capacity, and density, or it can be measured directly using the thermal lens technique [2]. This work aims to determine the thermal diffusivity of silver nanofluids on concentration using thermal lens spectroscopy. Silver nanofluids were fabricated using a variation of Turkevich's method, where silver nanoparticles were synthed from the reduction of silver nitrate by excessive use of sodium citrate [3]. The thermal lens experiment was performed using the double-beam setup in mismatched mode. The obtained results of the thermal diffusivity of silver nanofluids were of the order of 10-7 m^2/s which are in agreement with the results reported in the literature [2].

References:

[1] Younes, Hammad, et al. Nanofluids: Key parameters to enhance thermal conductivity and its applications. Applied Thermal Engineering, 118202 (2022).

[2] Lopes, C. S., et al. Determination of the thermal diffusivity of plasmonic nanofluids containing PVP-coated Ag nanoparticles using mode-mismatched dual-beam thermal lens technique. Advances in Condensed Matter Physics 2018, 1-6 (2018).

[3] J. Turkevich and P.C. Stevenson, A study of the nucleation and growth processes in the synthesis of colloidal gold, Discussions of The Faraday Society, 11, (1951).

Thermal sensitivity and structural changes of CaF_2 nanocrystals: xEr^{3+} (x = 0.5, 1.0, 1.5, 2.0 and 2.5%), synthed by hydrothermal and coprecipitation routes for optical thermometry

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In order to synthe materials with excellent properties for different applications, robust synthesis routes have been improved. Here, we studied and compared the luminescent properties and thermal sensitivity of calcium nanofluorides doped with 0.5, 1.0, 1.5, 2.0 and 2.5 of the $ErCl_3$reagent (% of mass in excess). The sample were synthed following hydrothermal and co-precipitation routes. For the structural and morphological analysis, transmission electron microscopy, XDR and scanning electron microscopy were carried out. In these analyzes, the formation of the face-centered cubic crystalline structure of $CaF_2$was confirmed through the Reitveld refinement method, and the average of the crystallite is calculated to be around 55 and 68 nm for co-precipitation and hydrothermal routes, respectively. Surfaces with little roughness and porosity were found. For the luminescence measurements, the samples were excited using a laser operating at 980 nm to obtain the upconversion emissions at visible region. Moreover, we used the optical thermometry measurements to obtain the thermal sensitivity via the luminescence intensity ratio technique of thermally coupled levels. The synthesis routes were discussed in terms of the efficiency of material for the application of these nanoparticles as nanothermometers for remote temperature measurements. These results show that these nanoparticles have excellent potential for application in nanothermometers [1].

Acknowledgments

Authors would like to thank FACEPE, for the financial supports, the PPEngFis at UFRPE and the IF at UFAL for their technical and laboratorial supports.

References

[1] S. Balabhadra, M.F. Reid, V. Golovko, J.P.R. Wells, Influence of the synthesis method on preferential clustering of Yb^{3+}in CaF_2:Yb^{3+}/Er^{3+}upconverting nanoparticles, Opt. Mater. 112 (2021) 110736. https://doi.org/10.1016/j.optmat.2020.110736.

Two-step laser ablation for the synthesis of Au-Ag alloy nanoparticles and their functionalization for nanophotonics applications

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Designing and synthesizing nanomaterials with controlled properties is a critical challenge within nanoscience and nanotechnology. In particular, nanoparticles (NPs) have expanded into diverse areas ranging from light energy harvest in third-generation solar panels to biomedicine. The synthesis of NPs is usually done by chemical reduction methods that must be specifically designed for each type of material. These chemical routes unavoidably lead to unreacted precursors and additives that tend to remain in the final colloidal product, therefore, requiring vast efforts for purification. Laser ablation synthesis in liquid (LASiS) is an alternative approach for nanoparticle generation, for it allows the production of colloids directly from nearly all classes of materials in pure solvents. Considering these highly pure NPs present promising potential, in this work, we present a practical example in which hybrid Au-Ag NPs are synthesized in a fast, high-yield, two-step LASiS method, thus becoming readily available for plasmonic-based applications, such as Raman/SERS biosensors and light improved absorption in organic solar cells. Our proposed synthesis combines LASiS with 1064 nm (400 ns; 0,5 mJ) pulses from a fiber laser at 50 kHz, leading to pure Au and Ag colloids. These colloids are mixed and submitted to a second irradiation with 532 nm (5 ns; 100 mJ) pulses from a Nd:YAG laser at 10 Hz. By varying experimental conditions, we mapped the kinetics of Au-Ag alloy formation through surface plasmon resonance (UV-Vis) measurements and particle size (DLS) determination. We also investigated Au-Ag alloy NPs interaction with biological molecules containing amine, thiol, and carbonyl functional groups, a significant step for the conjugation of biomolecules such as aptamers and antibodies to create highly specific and sensitive biosensors. In addition, the broader plasmon band manifested in the alloy NPs suggests their potential for light energy harvest applications.

Understanding the RE1-xNbx–BTC (RE: Eu or Tb) MOFs obtention by femtosecond laser ablation in liquid environment

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Metal organic frameworks (MOFs) is an important class of highly porous hybrid materials. It has been synthed by many methodologies like solvothermal, hydrothermal, sol-gel and more recently, by laser ablation in water environment [1,2]. High temperatures and pressures can be reached by cavitation bubbles from laser ablation in liquid environment (PLAL) but the phenomena stages are huge different when conduced with nanosecond or femtosecond lasers [3]. Although, studies of MOFs synthesis by femtosecond laser ablation are rare orinexistent. The aim of this work is to
investigate the obtention MOFs based on europium, or terbium, and benzene-1,3,5-tricarboxyic acid (BTC), introducing niobium as a dopant to these structures, and investigate they optical and structural properties. The experiments consist to ablate the powders of Eu2O3, or Tb2O3, in DMF/water solution of BTC using a second harmonic (λ = 532 nm) of a pulsed femtosecond Nd:YAG laser. The scanning electron microscopy (SEM) presented needles structures to ER-BTC and X-ray diffractometry showed reminiscent peaks of oxides on powder obtained. Furthermore, the samples were analyzed by FTIR, Raman, TGA and Luminescence measurements.

Acknowledgments

This work was supported by: CNPq, CAPES and FACEPE.

References

[1] da Costa, O. M. M. M., De Azevedo, W. M. (2016). J. Lumin., 170, 648-653.
[2] Ataei, F., Dorranian, D., Motakef-Kazemi, N. (2020). J. Theor. Appl. Phys., 14, 1-8.
[3] Hamad, A. H. (2016). London, UK: IntechOpen.

X-Research on 2D Materials

Bottom-up fabrication of graphene nanoribbons: from ultra-high-vacuum to device integration

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Graphene nanoribbons (GNRs) show exciting properties deriving from electron confinement and related band gap tunability[1]. The ability to tune GNRs' electronic and magnetic properties at the single atom level makes them an ideal platform for a wide range of device applications, from classical transistors to spintronics. In this talk, I will give an overview of the necessary steps to bring GNRs from ultra-high vacuum (UHV) to device integration focusing on the synthesis, characterization, and transport measurements of atomically-precise graphene nanoribbons. After the UHV bottom-up growth, GNRs are transferred using different transfer methods based on wet processes, such as polymer-free[2]and an electrochemical delamination method[3], as well as semi-dry/dry-transfer methods. Those processes allow the characterization of GNRs fingerprint modes via Raman spectroscopy[3,4]and the characterization of their electronic properties on decoupled substrates such as quasi-free-standing graphene. Next, I will show our progress on integrating different armchair GNRs (5-, 9-, 17-AGNRs)[5,6]into field-effect transistors with different gate and contact configurations. As a brief overview, we recently demonstrated the highest Ion current GNR-FET device to date by using a double-gate configuration[7]. 9-AGNR-FETs showed I_{on}currents up to 12μA and I_{on}/I_{off}up to 10^5. By integrating 9-AGNRs into FET devices using graphene[8]and carbon nanotubes[9]as electrodes, we also demonstrated tunable multi-gate devices showing quantum dot behavior with rich Coulomb diamond patterns.

1. Cai ***et al.***, Nature, 466, 2010
2. Borin Barin ***et al.***, ACS Appl. Nano Mater., 2, 2019
3. Overbeck & Borin Barin ***et al.***, Pssb, 12, 2019
4. Overbeck & Borin Barin ***et al.***, ACS Nano, 19, 2019
5. Llinas ***et al.***, Nature Comm., 8, 2017
6. Borin Barin ***et al.***, Small, 18, 2022
7. Mutlu ***et al.***, IEEE, 37.4. 1-37.4. 4, 2021
8. Zhang ***et al.***, Adv. Electron. Mater., 2201204, 2023
9. Zhang ***et al***, Nature Electronics, just accepted, 2023

Contributions of the Laboratory of Advanced Materials Chemistry – LAQMA-UFPR in layered materials, emphasizing layered double hydroxides and hydroxide salts

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Layered or two-dimensional (2D) materials belong to an important class of compounds with several industrial applications, to which 2 Nobel Prizes were attributed: 2010 - Physics - A. Geim, K. Novoselov - Graphene and 2019 - Chemistry - J.B. Goodenough, M.S. Whittingham, A. Yoshioro - Lithium batteries. After some contributions of Brazilian researchers, in 1992 was founded in the Federal University of Paraná, the LQES - Laboratory of Solid State Chemistry, nowadays LAQMA - Laboratory of Advanced Materials Chemistry. As far as the author know, the first works in Brazil involving exfoliation of MoS2 and use of the single layers in polymer nanocomposites were developed in this laboratory by the Scientific Initiation students [1,2]. These works started after the publication of the first synthesis of the metallic 1T-MoS2 in 1992 by the same author [3]. Probably the first atomic resolution image of 2H-WS2 using STM was published in a Brazilian Journal in 1998 [4], after the development of the STM technique just in 1986. Due to the experimental difficulties, lack of students and financial support, LAQMA was redirected to the synthesis, characterization, and application of Layered Double hydroxides - LDH and probably for the first time inBrazil, Layered Hydroxide Salts - LHS [4]. The objective of the present talk is to describethis evolution and the contributions of LAQMA in the scenario of layered materials in Brazil, emphasizing LDH and LHS, despite the scarce financial support since 1992, when the activities in the laboratory began. Future perspectives will also be addressed. Acknowledgements: This study was financed by CAPES - Finance Code 001, CNPq (FW: 300988/2019-2) and FINEP. References [1] Wypych, F., Seefeld, N., Denicoló, I., Quimica Nova., 20, 356 (1997). [2] Wypych, F., Adad, L.B., Grothe, M.C., Quimica Nova., 21, 687 (1998). [3] Wypych, F., Schöllhorn, R., J. Chem. Soc. Chem. Commun., 1, 1386 (1992). [4] Wypych, F., Weber, T., Prins, R., Quimica Nova., 21, 5

FLEXIBLE ELECTRONICS BASED ON CONJUGATED POLYMERS AND 2D MATERIALS

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The combination of conjugated polymers, oxides and carbon nanostructures can be an interesting way of organizing the nanostruture of thin films and develop new optoelectrical properties. The development of electronic devices based on thin films as electrodes or active layers offer some processing advantages and new possibilities in the manufacture of these devices, such as flexibility and large areas. The interest in this research area has grown significantly, presenting many innovations, whether in the synthesis of new materials, in the understanding of optoelectronic properties or in new device geometries allowing the increase of their efficiencies. The solution processed devices can take the advantage of nanostructured inks to allow their fabrication using spin coating or slot die coating in flexible substrates. In this work, some examples of the combination of these materials in thin films will be presented in the fabrication of electronic devices obtained by simple mixture in a common solvent; generated by interfacial synthesi and by miniemulsion technique. In addition, examples of electronic devices such as gas sensors, active layers of solar cells, COVID optical sensors and electrodes obtained with this approach will be presented and discussed.

From Electrochemical Sensors to Microfibers: Advancements in Graphene-Based Technologies

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Graphene-based sensors have received significant interest due to their unique properties. These properties include high electrical conductivity and high surface area. The 2D structure of graphene allows all carbon atoms to be exposed to the surroundings, making it an ideal platform for adsorbing and detecting molecules, which leads to increased sensitivity. Graphene is also biocompatible, providing a favorable environment for the immobilization of bioreceptors, such as antibodies, enzymes, DNA, and aptamers [1]. Thus, these properties make graphene an appealing transducer material in biosensors. In this presentation, an overview will be provided of the fundamentals and applications of graphene and its derivatives, such as graphene oxide (GO), reduced graphene oxide (rGO), and laser-induced graphene (LIG), in the development of electrochemical sensors and microfibers [2]. The presentation will explore the development of high-sensitivity devices based on graphene field effect transistors (FETs) and low-cost LIG-based electrochemical sensors [3]. Furthermore, a straightforward method based on 3D flow-focusing microfluid devices will be demonstrated to produce GO microfibers, with good control of thickness and length. Additionally, the presentation will explore both thermal and microwave treatments that can obtain rGO microfibers with spectroscopic and electrical properties like CVD graphene. These promising candidates are suitable for the development of flexible FETs and microelectrodes.

Acknowledgments
MackPesquisa, INCT NanoVida, CNPq, Fapesp 2019/14949-9 and FINEP.

[1] Nguyen, E. P.; Silva, C. C. C. and Merkoçi, A. Nanoscale, 12, 19043, 2020.
[2] Rocha, J. F. et al. Nanoscale, 13, 6752, 2021.
[3] Scroccarello, A. et al. ACS Sens., 9, 598, 2023.

Layered Materials for Optoelectronics and Quantum Technologies

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Graphene and layered materials (LMs) have great potential in photonics and optoelectronics, where the combination of their optical and electronic properties can be fully exploited, and the absence of a bandgap in graphene can be beneficial. The linear dispersion of the Dirac electrons in graphene enables ultra-wide-band tunability as well as gate controllable third-harmonic enhancement over an ultra-broad bandwidth, paving the way for electrically tunable broadband frequency converters for optical communications and signal processing. Saturable absorption is observed as a consequence of Pauli blocking and can be exploited for mode-locking of a variety of ultrafast and broadband lasers. Graphene integrated photonics is a platform for wafer scale manufacturing of modulators, detectors and switches for next generation datacom and telecom. Heterostructures based on LMs have properties different from those of their individual constituents and of their three dimensional counterparts. These can be exploited in novel light emitting devices, such as single photon emitters, and tunable light emitting diodes. LMs have potential for quantum technologies, as scalable sources of single photon emitters (SPEs). Quantum emitters in LMs hold potential in terms of scalability, miniaturization, integration. Generation of quantum emission from the recombination of indirect excitons in heterostructures made ofdifferent LMs is a path with enormous potential. I will overview production, characterization and application of graphene and LMs, focusing on optical and quantum applications.

Magneto-optical properties of van der Waals heterostructures

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Transition metal dichalcogenides (TMDs) are attractive van der Waals materials for fundamental physics, and for the next generation of optoeletronics and spintronics devices. Magneto-optical spectroscopy is a useful technique to investigating spin-valley coupling and exciton physics in two dimensional (2D) materials. In particular, under perpendicular magnetic field, interesting phenomena can be observed such as valley Zeeman splitting and magnetic-field-induced valley polarization. Twisted van der Waals (vdW) heterostructures based on TMD materials exhibit periodic variations, leading to a new type of in-plane superlattice known as moiré superlattice/pattern which modifies considerably the opticaland magneto-optical properties of excitons in vdW heterostructures. Furthermore, 2D ferromagnetic/antiferromagnetic vdW materials are promising systems for a new generation of spintronics or opto-spintronics devices. Particularly, by combining 2D magnets with TMDs, there is the advantage to use magnetic proximity exchange effects for making TMDmaterials magnetic. Furthermore, the physical properties of these materials or based vdW heterostructures are very sensitive to the number of layers, applied gate voltage, strain, twisted angles between the layers, spin-dependent charge transfer, magnetic proximity effects and etc, giving unprecedented controllability for their optical/magnetic properties. In this talk, I will present our recent investigation on the impact of smooth/local strain, doping and moiré pattern on magneto-optical properties of TMD heterobilayers and 2D magnet/TMD vdW heterostructures.

NANOSTRATEGIES FOR MOLECULE ADSORPTION WITH BIOLOGICAL AND ENVIRONMENTAL INTEREST

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In this talk it will be presented results combining ab initio simulations and molecular docking to develop complex models associated with experimental results. Mainly, carbon nanomaterials of different dimensions will be explored, applied in systems of biological interest [1], such as drugs, proteins, etc. and of interest for environmental remediation, such as adsorption of pollutants in liquid media [2].

It is noteworthy that this composition of methods, via nanoarchitectures, allows exploring different properties of nanosystems, reducing the experimental cost and creating new technological applications.

In some studies, we used experimental data from collaborating groups to corroborate the theoretically obtained analyzes and allow for innovative results for the advancement of nanoscience and nanotechnology.

[1] Schultz, Júlia Vaz ; et al . Structural Chemistry, 2023 https://doi.org/10.1007/s11224-023-02135-x

[2] Bitencourt Leão, Mayara, et al Molecular Systems Design Engineering, 2023, https://doi.org/10.1039/D2ME00268J

Screening nanocatalysts for agrochemical neutralization: selective functionalization and effects of coverage, support, group and neighboring

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Agrochemicals and chemical warfare have a lot in common, since many belong to the class of organophosphates. Our group has been focused in combating organophosphates, seeking nanocatalysts for degradation and detection. We pursued site-selective mono and bifunctionalization of various nanomaterials, such as graphene oxide and carbon nanotubes, with thiol, imidazole, hydroxamate groups (covalently linked) and metallic nanoparticles (Ag, Ni). The catalysts were obtained as powder and thin film. For the bifunctionalization, two different groups were anchored on specific sites of the material: both carboxylic acid and epoxy sites for the graphene oxide. The catalysts are able to act as neutralizing agents for organophosphates, accelerating the process to less than 1 day, that otherwise could take millions of years. The bifunctionalized catalysts showed an interesting relationship between coverage degree, group identity and neighboring effects. The materials with the highest degree of functionalization were not the best catalysts, attributed to cooperative effects between neighbouring groups that promote multiple catalyses. In addition, a higher coverage by functional groups can disturb or even inhibit possible catalytic mechanisms, either by steric effect or non-ideal positioning of the groups. This finding was extremely important since it shows that the functionalization process pursued should be driven by the application wanted since not always what we expect as a better functionalization methodology will lead to the best response for a specific application. The catalysts also showed a dual function: could be used as sensors (surface enhanced Raman spectroscopy, electrochemical) for these toxic agents. In summary, indeed the strategic design of various nanomaterials seeking optimal catalysts opens a myriad of possibilities for achieving prominent catalysis, especially for promoting worldwide chemical security by combating agrochemicals and chemical warfare.

Synergetic enhancement of quantum yields ofmonolayer WS_2 by proximal metal gating

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Quantum yield (QY) is a crucial performance factor of the semiconductor to be considered as light-emitting photonic devices. While the structural defects are the main source of degrading the quantum yield of bulk semiconductors, two-dimensional semiconductors display the major channel of non-radiative decay of excitons due to strong interaction among the excitons even in moderate exciton density conditions, In this presentation, I willdiscuss some of the strategies that we developed to suppress or bypass the exciton interaction, which lead to enhancing the QY of monolayer WS_2, specifically the use of proximal metal plates or hBN substrate and applying out-of-plane electric field. [1-3]

[1] Y. Lee, et al, Nat. Commun. 12, 7095 (2021)
[2] Y. Lee and T. T. Tran, et al, ACS Photon. 9, 873 (2022)
[3] T. T. Tran et al., manuscript in preparation

2D Materials for Photovoltaic Energy Harvesting

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Two-dimensional (2D) semiconducting materials as active layers in photovoltaic devices is a subject that has attracted a lot of attention in the last years. Nowadays, the most employed materials for this kind of application are transition metal dichalcogenides (TMDCs), which are semiconductors with chemical configuration MX_2, where M is a transition metal such as Mo, W, and X is a chalcogen atom such as S, Se, or Te. However, many other 2D materials have also been proposed. As far as TMDCs are concerned, while significant attention has been given to single layer TMDCs, a limited number of works have addressed the few layer case which is particularly relevant for photovoltaic devices.

Herein, we studied the electronic and optical properties of few layer TMDCs composed of Mo, W, S, and Se within the GW and Bethe-Salpeter approach. First-principles calculations based on density functional theory were carried out using the Quantum ESPRESSO package. The many-body perturbation theory and Bethe-Salpeter calculations were performed using YAMBO code. We address the photovoltaic performance of these TMDCs estimating the spectroscopic limited maximal efficiency (SLME) as a function of the thickness of the semiconductor. We compared the different TMDCs to known materials used in photovoltaics paving the way for efficient nanoscopically thin solar cells.

We have also employed the same methodology described above to study a polymorph of h-BN that has been theoretically proposed recently. This material, named as orthorhombic diboron dinitride (o-B2N2), is a direct band gap semiconductor. We show that the band gap energy of o-B2N2 varies strongly with number of layers and consequently it has potential to be employed in photovoltaic devices.

3d transition metal coordination on monolayer MoS_2: a facile doping method to functionalize surfaces

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Earth abundant metals-based catalysts can be found as a good option for new catalysts with remarkable activity in comparison with Pt-based, as TMDs like MoS_2, $MoSe_2$ and WS_2 have shown to possess activity towards HER, mostly on the edge, and the basal plane with characteristic low activity. Manipulating the surface of these materials can be a good way to enhance the overall activity and activate the basal plane. Due to the nature of the dichalcogenide atoms, having free electron pairs acting as a Lewis base, one can coordinate metal atoms, like 3d transition metals ions, as a Lewis acid, creating new and stable functionalized materials [1]. In this work we show a facile way of functionalizing TMDs surfaces with 3d TM atoms. X-ray Photoelectron Spectroscopy (XPS) results indicate that depending on the doping atom, one can have a p or n-type doping, as Ni and Co species can donate electron density, although Cr and Cu can take electron density. Photoluminescence Spectroscopy shows by the Exciton/Trion ratio that a minimum amount of doping atom is sufficient for changing this ratio, indicating electron density changes. High Resolution-Transmission Electron Microscopy (HRTEM) demonstrates for Ni-doping that the atoms are atomically dispersed on the surface [2]. Therefore, the formation of TMD-TM complexes can lead to a controllable way of modulating the material’s properties.

Acknowledgements: Instituto Serrapilheira, CNPq, CAPES, FAPERJ.

References:

[1] S. Lei, Nat. Nanotechnol. 11(5), 465-471 (2016)
[2] W.Costa Silva and H.Liu, Nanoscale 14, 10801-10815 (2022)

CHARACTERIZATION AND QUANTIFICATION OF ZERO AND ONE-DIMENSIONAL DEFECTS IN WS2 BY RAMAN AND PHOTOLUMINESCENCE SPECTROSCOPIES

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In recent years, transition metal dichalcogenides (TMDs) have been widely studied due to their unusual optoelectronic properties. Recently, the study of defects in these materials allows the application in semiconductor devices, since defect sites locally disturb the crystalline lattice, leading to peculiar effects. For example, punctual defects (zero-dimensional) are the single-photon emission source, which is an important role in the quantum information devices industry [1], and highly ordered defect lines (one-dimensional) exhibit charge wave densities along themselves [2]. In this context, Raman and photoluminescence spectroscopies have been a powerful tool to quantify and identify punctual and linear defects in 2D materials. In this work, we synthesized monolayers of WS2 via chemical vapor deposition (CVD), bombarded these samples with He ions and we verified the existence of two Raman bands and two new electronic transitions, present in the photoluminescence spectra, activated by punctual defects. Linear defects were produced by lithography, also with He ions, and show promising results, such as the anisotropy of some Raman modes. First-principle calculations were performed to obtain the electronic and phononic dispersion, thus inferring the origin of the observed effects in Raman and PL spectra. The same punctual defect model that allows to predict the crystallinity of graphene and molybdenum TMD [3] was employed to visualize when WS2 begins to become amorphous. So, this work makes possible the identification of defects in tungsten TMDs, which are much used in the technology industry of semiconductors.

References
[1] HE, Yu-Ming et al. Nature nanotechnology, v. 10, n. 6, p. 497-502, 2015.
[2] MA, Yujing et al. Nature communications, v. 8, n. 1, p. 14231, 2017.
[3] MAIA, Fábio Cardoso Ofredi et al. Nanotechnology, v. 32, n. 46, p. 465302, 2021.

Defect engineering on the basal plane of ultra-largeMoS2 monolayers for electrochemical hydrogen production

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The basal plane of molybdenum disulfide (MoS2) monolayers displays the largest area available for electrocatalysis, however, due to the lack of active sites on this region the electrocatalytic activity toward electrochemical hydrogen production is poor [1]. Thus, many defect engineering routes have been used to tune the electrocatalytic activity of the basal plane to improve its performance. However, it is still very challenging to control the position, size, and propagation of defects on the basal plane of MoS2 monolayers by most of defect-engineering routes. Thus, in this work, we will show the fabrication of arrays of defective regions on supported and free-standing ultra-large MoS2 monolayers. First, ultra-large MoS2 monolayers were prepared directly on conductive substrates using a simple and fast electrochemical thinning method [2]. Next, gallium-FIB was used to etch MoS2 and open reactive windows on the basal plane. The dose applied to etch a controlled depth on MoS2 was calibrated using atomic force microscopy. The arrays were characterized using Raman spectroscopy and photoluminescence (PL) experiments, thus confirming the introduction of defects on the basal plane. The impact of each dose on the electrocatalytic activity toward hydrogen evolution reaction was measured by fabricating microelectrodes. TEM analysis also revealed the formation of amorphous regions. In addition, we will show the fabrication of arrays of etched edges using O2 plasma and electron-beam lithography.

Acknowledgements: Serrapilheira Institute (1912-31228), FAPESP (2022/00955-0).

[1] L. H. Hasimoto, J. Bettini, E. R. Leite, R. S. Lima, J. B. S. Junior, L. Liu, M. Santhiago, ACS Applied Engineering Materials 1, 2, 708-719 (2023)
[2] N. Freitas, B. R. Florindo, V. M. S. Freitas, M. H. de O. Piazzetta, C. A. Ospina, J. Bettini, M. Strauss, E. R. Leite, A. L. Gobbi, R. S. Lima, M. Santhiago, Nanoscale, 14, 6811 (2022)

Doped 2D diamond: properties and potentialapplication

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Graphene is the most popular two-dimensional (2D) material, being a zero-gap semimetal with a honeycomb carbon structure and sp^2 hybridization. It is the fundamental building block for several other nanomaterials, such as fullerenes, nanotubes, graphite, and the single-layer diamond (2D diamond). Furthermore, the properties of carbon-based materials could vary considerably by doping and/or functionalizing them with several distinct types ofatoms and by combining them with different 2D systems [1]. Due to recent developments inthe synthesis and characterization of 2D materials, the 2D diamond has received great attention, with promising applications in several fields, such as batteries, quantum computing, nano-optics, and nanoelectronics. In this context, we explored the structural, thermodynamic, dynamic, elastic and electronic properties of C_4X_2 (X = B or N) systems. These structures are made of three graphene sheets in which four C atoms are bonded covalently (2D diamond-like) in each unit cell. The two external layers are 50% doped with X substitutional atoms and, hence, each X atom is bonded to three C atoms. We found that the C_4X_2 systems present stiffness comparable to that of the diamond. Additionally, the C_4N_2 nanosheets have wider indirect bandgaps, while the C_4B_2 systems have narrower direct bandgaps when compared with the pristine 2D diamond. These results provide chemical routes to tune the electronic properties of 2D diamonds by doping them for specific applications, such as nano-optoelectronic.

Acknowledgements:

This investigation was partially supported by Brazilian agencies CNPq, CAPES and FAPESP (22/10095-8).

References:

[1] Ipaves, B., Justo, J. F., and Assali, L. V. C., J. Phys. Chem. C 123, 23195 (2019).

Electrical control of excitonic complexes in $MoSe_2$ homobilayers

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The twist angle between the monolayers directly controls the effects of periodic moiré potential in transition metal dichalcogenides (TMDs) bilayers [1]. Nonetheless, such effects may appear even more pronounced at twisted homobilayers, since the absence of lattice mismatch promotes the appearance of larger Moiré superlattices(ref) and reconstructed domains. Novel features arising from intra- and interlayer excitons, such as their ultrafast formation and charger transfer, long population recombination lifetimes, and binding energy of ~150 meVs[2,3], turn TMD hetero-structures into an attractive device for the study and manipulation of optical and transport prop-erties via electrical fields and electrostatic gating[4].

In this work, we explore the ability to control excitonic complexes in MoSe2/MoSe2 twisted bi-layers (t-BL) by performing gate-dependent microphotoluminescence (μPL) spectroscopy at room temperature. We observe the energy tunability of several meVs occurring at the emission of excitonic complexes derived from the t-BL region compared with MoSe2 monolayer response. More-over, we investigate the twisted-angle dependence of valley properties by studying two hetero-structures that present distinct stacking angles, 0 degrees (R-stacking) and 18 degrees. Our results suggest that the two prominent interlayer exciton species in the R-stacking t-BL, which can be individually manipulated, may be associated with the moiré superlattice formation. Although our findings still require further understanding, we present an electrical control of excitonic complex-es at room temperature, poorly explored in the literature of TMD hetero-and homobilayers, and open up new pathways to survey applications in semiconducting optoelectronic field.

[1] K. L. Seyler et al., Nature 567, 66-70 (2019)
[2] A. Ciarrocchi et al. Nature Photonics 13, 131 (2019)
[3] H. Baek et al. Science Advances 6, 37 (2020)
[4] T. Andersen, Nature Materials 20, 480 (2021)

Electrons, ions and photons as tools for modify andstudy graphene and related materials

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Graphene, a two-dimensional sp2 carbon structure, is a system that comprises exciting possibilities to demonstrate new physics and promising applications, many of which can be appreciated only by means of nanoscale modifications in its structure. In this talk, we will journey through the use of nanotechnology tools to understand the behavior of this material and also, tune its properties for desired applications. It will be discussed the metrology of defects and disorder in graphene and the use of focused ion beam milling, i.e. Ga (Focused Ion Beam - FIB) or He (Helium Ion Microscope - HIM) [1], in the fabrication of periodical patterns and devices. Also, we are going to show our work on revealing lattice disorder, oxygen incorporation and pore formation in laser induced two-photon oxidized graphene [2]. Our work is developed using FIB, HIM, high resolution transmission electron microscopy (HRTEM), electron energy loss spectroscopy (EELS), atomic force microscopy (AFM) and Raman spectroscopy. Finally, we are going to show our efforts in optical characterization at resolutions bellow the limit of light diffraction through the use of tip enhanced Raman spectroscopy (TERS) [3-5].

[1] Archanjo, B. S. et al. Appl. Phys. Lett. 104 (2014)193114.
[2] Mendoza, M. E., Archanjo, B. S. et al. Carbon 143 (2019) 720.
[3] Archanjo, B. S. et al. ACS Photonics 5 (2018) 2834
[4] Vasconcelos, T. L., Archanjo, B. S. et al. ACS Nano 9 (2015) 6297.
[5] Vasconcelos, T. L., Archanjo, B. S. et al. Ieee Journal of Selected Topics in Quantum Electronics 27 (2021) 4600411

Evolution of nanoRaman & Correlative Spectroscopy for 2D Materials: Innovative Sample scanning, Automatic Chemometrics, Imaging, Colocalized Correlations with Scanning Electron Microscopy, X-ray Microfluorescence & Others Spectroscopy Techniques.

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This presentation will focus on Raman, NanoRaman and Correlative Spectroscopy applied to Materials Science and Engineering of Nanomaterials. The principles of applied Raman spectroscopy and Raman imaging (the importance of spectral resolution for imaging) will be approached from theory, application and instrumentation as the main topics for two-dimensional (2D) materials characterization for a wide range of applications such as biomaterials and life sciences. Raman and nanoRaman Spectroscopy are one of the only techniques capable of providing precise and non-destructive analysis combined with high resolution images. Raman spectroscopy provides valuable information such as chemical and structural composition. Based on the light-matter interaction, the Raman technique can perform characterizations through mapping measurements, obtaining relevant information, such as: particle size distribution, homogeneity, grain size, phase changes and various other characteristics of the sample through the chemical evaluation of the material. The effects of chemical bonding, strain, and crystal size will be covered, and we will show how to measure and image these features. In addition, we will discuss combined laser-excited photoluminescence and Raman scattering images of two-dimensional (2D) crystals to reveal the spatially variable solid-state structure of these nanomaterials. The development of the instrumentation allows the syllabic separation of Raman spatially co-localized and correlated with other techniques, such as Photoluminescence, Fluorescence, Scanning Electronic Microscopy and Atomic Force Microscopy, being able to reach resolutions on the nanometric scale. The techniques can be applied in several areas of knowledge, such as pharmaceuticals, photovoltaics, graphene, cells, nanoparticles, nanoplastics, among others.

Exploring the use of earth-abundant layered materialsin 2D-based studies

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Phyllosilicates minerals are an emerging class of naturally occurring layered insulators with large bandgap energy that have gained considerable attention from the scientific community. This class of materials has been explored recently at the ultrathin two-dimensional level due to their specific mechanical and optoelectronic properties, which are crucial for engineering novel devices. Phyllosilicates have lamellar structures and can be embedded into van der Waals heterostructures due to the possibility of exfoliation down to monolayers and easy handling. Due to these properties, phyllosilicate minerals can be considered promising low-cost nanomaterials for electronics, photonics, and optoelectronics future 2D-based device applications. Here, we present features of these nanomaterials that are relevant to their use in potential 2D-based applications [1-3], discussing the major challenges in working with them.

Acknowledgments:

Fundo Mackenzie de Pesquisa e Inovação (No. 221017), FAPEMIG, CAPES, CNPq, and FAPESP (No. 2020/04374-6).

References:

[1] Barcelos I D, et. al., J. Appl. Phys. Submitted (2023)

[2] de Oliveira R, et. al., J. Opt. Soc. Am. A 40, 153959 (2023)

[3] Cadore A R, et. al., 2D Mater. 9, 035007 (2022)

Film formation and chiro-dependent properties ofchiral 2D perovskites

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Organic ammonium-based cations are used to prepare low-dimensional perovskite structures, allowing fine-tuning of perovskite's optical and electronic properties. Recently, chiral organic cations have been introduced to produce low-dimensional perovskites. The asymmetric hydrogen (H-) bonding between the H in the ammonium group and the halogen in the perovskite structure, breaks the centrosymmetry of the crystal, resulting in chiroptical activity in 2D perovskites. Despite the growing interest in chiral perovskites, the fundamental process underlying their crystallization and film formation remains unknown. For example, 1D phase impurities in 2D chiral perovskite systems are often reported, but strategies to circumvent this issue are still lacking. In this study, we investigate the formation of 1D phases in 2D perovskites based on chiral methylbenzylammonium cations and their racemic mixture. By combining in- and ex-situ characterizations, we demonstrated that the solvent choice, annealing condition, and spatial configuration of the organic cation play a significant role in determining the final film's microstructure, phase purity, and crystalline orientation. We propose that the 2D and 1D phases are the kinetic and thermodynamic products of the film formation reaction, respectively. The film formation dynamics are shown to be influenced by the solvent's coordination ability, allowing strategies to control phase purity in chiral 2D perovskites. We also find that the chirality of the organic cation and film processing condition impacts the degree of energetic disorder, residual lattice strain, and in-plane conductivity of the films. This work provides relevant insights for the development and future integration of chiral perovskites into optoelectronic and spintronic devices.

Acknowledgments: FAPESP (2017/11986-5, 2018/21401-7, 2020/04406-5, 2021/12104-1, 2021/01357-6, 2018/01669-5, 2014/25770-6). DFG (SPP 2196 #424216076, KO6414)

First principles study of ZnX and CdX (X=S, Se, Te)direct band gap semiconductors and its van der Waals heterostructures.

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2D van der Waals heterostructures have emerged as an important class of materials due to their unique properties, which are derived from the combination of different 2D materials with complementary properties. These heterostructures allow for the creation of complex electronic and optoelectronic devices with unprecedented functionality and performance. This work presents a comprehensive first principles study of ZnX and CdX (X = S, Se, Te) direct band gap semiconductors and their van der Waals heterostructures. The electronic and optical properties of these semiconductors were analyzed calculations revealed that ZnX and CdX (X = S, Se, Te) are all direct band gap semiconductors. Furthermore, the electronic band structures and density of states were calculated for the studied semiconductors, and the results showed that ZnX and CdX semiconductors have similar electronic structures, with the conduction band minimum (CBM) and valence band maximum (VBM) located at the Γ point in the Brillouin zone. However, the width of the band gap and the effective mass of the charge carriers vary with the type of semiconductor.The study also investigated the electronic and optical properties of van der Waals heterostructures composed of ZnX and CdX semiconductors. The heterostructures were formed by stacking the different layers of the semiconductors with different orientations. The results show that the band alignment at the interface between the two semiconductors depends on the type of semiconductor and the stacking configuration. The heterostructures exhibit interesting optical properties, such as type-II band alignment, which is attractive for optoelectronic applications.The results obtained can be useful in the design and development of novel semiconductor devices and materials for optoelectronic and photovoltaic applications.

Grafeno Brasil Platform: a booster for consolidation of graphene-based products' market in Brazil

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The "Grafeno Brasil" network is particularly well suited for companies and institutions working in the field of graphene research and development. The network''s main goal is topromote innovations in Brazilian industry through the development and placing on the market new graphene-based products that meet the needs of customers, on the one hand,and all regulatory requirements, on the other hand. "Grafeno Brasil" platform puts on itsbasis the principles of demand-side innovation policy with the introduction of feedback linkages between supply and demand in the innovation process. One of the key features of "Grafeno Brasil" network is its ability to match the demands (including unmet or latentdemands) of companies for innovation with the available capacities and resources of research institutions and, in general, of public sector. This approach to innovation by supporting the demand side, including utilization of demand-side instruments such as regulation and standardization, is supposed to create more efficient and effective collaborations between all actors, reducing time-to-market for new products and technologies. Through its on-line platform, the Grafeno Brasil network will provide a variety of tools and resources to facilitate communication and collaboration between its members, as well as to help forward the coordination between the regulators and the different stakeholders. In conclusion, the Grafeno Brasil network is the innovative and dynamic platform, which is designed to help diffusing innovation and fostering collaboration between companies and research institutions. Its unique approach to matching the demands and capacities creates new opportunities for growth and development in various industrial sectors and business areas, as well as promotes the advancement of cutting-edge technologies such as graphene-based technologies.

Acknowledgments: CNPq, FAPERJ and Finep.

Graphene as a gate-tunable window for near-fieldimaging in the terahertz

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The well-established two-dimensional (2D) material graphene is anticipated to feature extraordinary near-field optical properties in the terahertz (THz) frequency range [1]. However, the experimental interrogation of such phenomena is non-trivial due to the intrinsic dimensionality mismatch between atomically thin graphene and the wavelength of the THz radiation reaching hundreds of micrometers. This task becomes even more challenging in the THz gap (0.1 - 10 THz) wherein powerful light sources are scarce [2]. Nevertheless, modern nanoscopy techniques coupled to unique, highly brilliant THz light sources have been demonstrated to accomplish an experimental investigation of such 2D materials [3]. In this work, we apply a scattering-scanning near-field optical microscope (s-SNOM) illuminated by THz radiation of a tunable free-electron laser (FEL) to investigate the near-field optical response of graphene within the THz gap (1.5 – 6.0 THz). We firstly demonstrate that graphene takes on an optically metallic character at around 2 THz. In this frequency range, the driving THz field enables quasi-static response associated to the relaxation of the Gr charge carriers. Secondly, we find the graphene to feature a distinct field-enhancement effect (FEE) at 3.8 THz, meaning a significantly increased optical contrast and sharpness of the near-field optical images. Through theoretical analysis based on the polariton dispersion relation and near-field modeling, the FEE can be attributed to a plasmonic resonance of graphene near 3.0 - 6.0 THz. We believe these novel aspects of graphene nanooptics in the THz range to prove valuable for future graphene-device-based studies and technological applications.

How near-field infrared microscopy enables vibrational analysis of natural 2D nanocavities for water confinement

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Water is the matrix of life and its confinement in nanocavities is a central topic from geophysics to nanotribology. Phyllosilicate layered minerals are natural nanocavities for water due to their capacity to hydrate by confining water molecules in interlamellar space. However, the hydration of phyllosilicates at the nanoscale is not a fully understood process and depends on the geological specimens. In order to obtain information about the confinement of water in phyllosilicate minerals, we can investigate the vibrational properties of these systems, since water molecule has three vibrational modes active in infrared (IR). However, accessing the vibrational assignment of an isolated few-layer (FL) phyllosilicate flake through far-field analysis in IR absorption experiments is extremely challenging. Limited by diffraction, these experiments are unable to reach nanometric resolution since the wavelengths of IR radiation are micrometric. Evidently, searching for the vibrational response of few water molecules confined in this two-dimensional (2D) system is even more challenging. In this perspective, scattering-type near-field optical microscopy (s-SNOM) and synchrotron infrared near-field spectroscopy (SINS) are key toolsto reach sub-diffractional nanometric resolution in the study of 2D systems.

In this presentation, we will provide technical aspects that make s-SNOM and SINS powerful techniques in nano-optics and local chemistry studies of a variety of natural 2D phyllosilicates [1]. We will demonstrate SINS as a robust alternative to perform opto-vibrational characterization of FL-phyllosilicates by enabling local nanospectroscopy. As unique result, we will unravel the water confined in an ultrathin phyllosilicate flake by IR s-SNOM nanoimaging. This result provides a way to access the outstanding capacity of phyllosilicate minerals to absorb water and act as natural nanocavities that can be explored in future technologies.

[1] https://doi.org/10.1364/JOSAA.482518

Intrinsic Mechanical Strain in Chemical VaporDeposition-Grown MoS2/MoSe2 Monolayer Heterostructures

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In this work, we have synthed 2D MoS2/MoSe2 heterostructures by chemical vapor deposition (CVD) in a one-pot, two-step configuration: the second crystal was directly grown on top of the first, without mechanical transfer. Indeed, most vertically stacked HSs go through nanomechanical manipulations resulting in nonhomogeneous residual strain, polymer residues or mechanical fracture. Also, among others, an advantage of such heterostructures is that they can be considered as pristine vertical HS, since they have a clean interface between layers. Moreover, another interest in studying such material is that the bottom and top layers are almost the same. They only differ by the chemical nature of the chalcogen atom, and consequently the of the metal-chalcogen bond is slightly different. The photoluminescence and vibrational analyses indicate shifts in the band positions when compared to monolayer MoS2 or MoSe2. We propose that this behavior is mainly driven bya mechanical strain that originates from the synthesis, since the HSs tend to counterbalancethe lattice mismatch between the top and the bottom crystals. Our findings are corroboratedby DFT calculations, where electron and phonon dispersions of strained materials reproduce qualitatively well the experimental data. Also this work permits us to compare the interaction between constituent layers of HS assembled by transfer or by direct growth and their role to tune the physical properties of those nanomaterials for specific applications in nanoelectronics.

[1] W. S. Ramos et. al. ACS Appl. Nano Mater. 2023, 6, 14, 13342-13351.

Light emission from epsilon near zero resonances inpolar 2D crystals

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The electromagnetic field can exhibit distinct structures in the far and near-field radiation zones [1]. Aspects of such peculiar behaviors have been seen from the study of high momenta polaritons observable only in the near-field zone of polar two-dimensional (2D) crystals [1] like hexagonal boron nitride (hBN) and alfa molybdenum trioxide (αMoO_3). It is well documented that such polaritons exist in the range between the transverse (ω_{TO}) and longitudinal (ω_{LO}) optical phonons, wherein the real part of the media's electrical permittivity (ε) is negative. However, close to such spectral limits, the polaritonic activity vanishes, whilst other interesting near-field phenomena rise as fascinating optical effects found close to ω_{LO} when $\varepsilon \to 0$, which creates the epsilon near zero (ENZ) spectral region. Decoupling of magnetic and electric fields in Maxwell equations, infinite impedance, slow-light and super transmission/reflection [2, 3] are exquisite effects originated from the ENZ condition. In this presentation, we will show that 2D hBN and αMoO_3 emit light in the near-field zone, in ENZ bands where $1 \leq Re[\varepsilon] \leq 0$, due to ENZ resonances of the local density of states. Such discoveries are experimentally recorded by real-space nanoimaging in the mid and far-IR using a scattering-scanning near-field optical microscope coupled to quantum cascade lasers and a free electron laser. Proper discussion on the physical implications is given from theoretical predictions. Supported on quantitative convergence of experiment and theory, we demonstrate that ENZ resonances are general effects of 2D polar hyperbolicmedia that can be further controlled by temperature, strain, and electrical gating.

Acknowlegments:

We thank the Brazilian Synchrotron Light Laboratory and the Free Electron Laser of Dresden, Germany, for support in the experiments.

References:

[1] Low, T. et al., Nat. Mat. 16, 182 (2017).

[2] Liberal, I. et al., Nat. Phot. 11, 149 (2017).

[3] Alù, A. et al. PRB 75 155410 (2007).

Luminescence enhancement of CVD grown Ga-dopedWS_2 monolayers

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Monolayer Tungsten disulfide (WS_2) is a direct band gap semiconductor that boasts excellent luminescence properties, which may be useful in many optoelectronics applications. Through the incorporation and control of impurities or defects in the lattice of WS_2, one can significantly alter a specific property to improve the performance of devices for a target application, such as photonics and catalysis. In this study, we utilized in situ doping strategy to synthe Ga-doped WS_2. Our results indicate that Ga- doped WS_2luminescence is enhanced by nearly four times when compared to pristine WS_2. To confirm the incorporation of Ga in WS_2structures, Resonance Raman spectroscopy and X-ray photoelectron spectroscopy (XPS) were utilized as characterization methods. Raman spectroscopy revealed that the presence of Ga affects the crystals' vibrational properties, where disorder-related effects due to a higher density of defects are directly affected, where the LA(M)/[A_{1g}(M)-LA(M)] peak intensity ratio increased due to the presence of Ga impurities in the lattice. In particular, the small redshift observed in the A1g mode indicates that Ga species are n-doping the monolayers, which is compatible with the literature results seen elsewhere. Also, from the XPS spectrum, it is clear to observe an energy blueshift in W4f and S2p regions in Ga-doped WS_2compared with pristine WS_2. Especially, there is an evolution of blueshift in Ga2p region with increasing Ga dopant. These results prove the presence of Ga in the WS_2lattice.

Magneto Dependence of Excitonic States of TwistedWSe2

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Monolayer transition metal dichalcogenides (TMDs) exhibit rich physics related to their excitonic complexes. In these materials, the reduction of the dielectric screening collaborates with the intensification of Coulomb interaction and, consequently, the formation of exciton with high binding energy and other complexes involving more carriers. In particular, TMDs present spin-orbit coupling, promoting valence and conduction band splitting, and valley structure can be verified with circularly polarized light. In a slightly different system, the band structure and the excitonic properties can be tuned if two layers of TMDs are placed, one on top of the other with a twist angle, producing a twisted WSe2 material (t-WSe2). In our work, we investigate the excitonic states of high-quality t-WSe2 encapsulated in hBN crystals. We investigate the photoluminescence''s polarization and magneto dependency at low temperatures. Comparing with the monolayer case, we observe unexpected high valley polarization for the exciton and negative biexciton. Moreover the similar effect was measured for other unidentified excitonic complexes with energies closed to reported values for dark trions and local excitons, which generally shows small valley polarization. Finally, we observe a new emission that rises for high magnetic fields (B >7 T) is verified whose identification is still under progress. Our results may reveal the quantum confinement effects of Moiré excitons in twisted semiconducting 2D heterostructures.

Acknowledgments:

This work was supported by CNPq, INCT/Nanomateriais de Carbono, CAPES and FAPEMIG, Brazil. The authors are thankful to the Laboratory LCPNano of UFMG.

Multilayer Graphene as an Endoreversible Otto Engine

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Graphene is a remarkable material, belonging to the class of Dirac materials whose electronic structure gives rise to charge carriers that behave as massless fermions, exhibiting relativistic behavior at the laboratory scale. This makes graphene an ideal system for exploring the thermodynamics of relativistic quantum systems. In particular, the energy spectrum of multilayer graphene structures, subjected to an external magnetic field, depends strongly on the number of layers. To investigate the performance of a finite-time endoreversible Otto cycle with multilayer graphene systems as working media, we analyze the engine efficiency as a function of the number of layers. Remarkably, we find that the efficiency at maximum power can exceed that of a classical working medium, with the bilayer system showing the optimal performance. Overall, our results shed new light on the fascinating thermodynamics of graphene multilayers and open up exciting possibilities for its use in advanced technologies.

References

[1] S. Deffner, Efficiency of harmonic quantum otto engines at maximal power, Entropy 2018, 20, 875

[2] Abe, S. Maximum-power quantum-mechanical Carnot engine. Phys. Rev. E 2011, 83, 041117.

[3] Nathan M Myers, Francisco J. Peña, Natalia Cortés and Patricio Vargas, Multilayer Graphene as an Endoreversible Otto Engine. https://arxiv.org/abs/2212.03286

New 2D materials from non-van der Waals solids

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The advent of graphene created a revolution in materials science and renewed the interest in new 2D materials. Up to now, most of the 2D structures were obtained from lamellar solids (the so-called van der Waals solids). Because there are no strong bonds among the layers, the structures are easily exfoliated. Recently [1], the first 2D material obtained from non-van der Waals solids (hematene, obtained from hematite) was reported, which opened the perspective to obtain a large number of new 2D materials. Hematite was followed by ilmenite, chromite, magnetite, pyrite, all, 2098mong others [2], as 3D structures used to create new 2D ones. More recently [3], 2D structures from silicates were reported. In this work, we discuss (from theoretical and experimental views) the structural and electronic properties of the new 2D materials, as well as their potential applications as sensors and energy harvesting.

Acknowledgments:
Work was supported in part by the Brazilian agencies CNPq and FAPESP. [1] A. P. Balan et al., Nature Nanotech. 13, 602 (2018).
[2] A. P. Balan et al. Mater Today 58, 164 (2022).
[3] P. R. Mahapatra et al., J. Mater Chem. C 11, 2098 (2023).

Novel two-dimensional materials from abundant minerals: adding value and strategic applications

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Nothing short of a scientific-industrial revolution is expected from two-dimensional (2D) materials thanks to their exceptional properties that can change paradigms from Engineering to Nanomedicine. In order to become a reality, new accessible 2D materials with different properties will be needed. Therefore, the main focus of the present work is the use of earth-abundant minerals as precursors of new 2D materials.

Our team tackles three main challenges: (1) the prospection of reasonable candidates and their exfoliation mechanisms (focusing on liquid phase exfoliation, which can be scaled up); (2) the characterization and understanding of the physical properties of the new materials (isolated, in heterostructures or composites), using a theoretical-experimental methodology that combines computer simulations with microscopy and spectroscopy technique and (3) the application and validation of the most promising minerals for the production of advanced biomaterials. The theoretical-experimental characterization of the 2D flakes produced (and their possible associations with other materials and molecules) enable the understanding of their new properties and, consequently, the proposal of scientific-industrial applications.

We will illustrate this approach with several examples, from talc to gipsite, including other 2D materials from abundant minerals. In this sense, talc was out first proposal [1] and is the most advanced towards a real-world application as part of an advanced biomaterial [2, 3]. Just like graphene, produced from natural graphite and with a range of spectacular properties different from the precursor mineral, the present work has the potential to add value to abundant minerals and reveal new pillars for the coming revolution.

References:
[1] Ananias B Alencar et al. 2D Mater. 2 015004 (2015).
[2] Laureana M Mota et al. Surf. Coat. Tech 397, 126005 (2020).
[3] Ana Barbara Batista et al. Surf. Coat. Tech. 441, 128589 (2022).

Optical Study of Graphene/Water Interactions

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Among all the 2D materials, graphene is the most studied, it is the first isolated 2D material, and further to its atomically thin structure, it also presents admirable electrical, thermal, and mechanical properties. Those properties motivate using graphene as a membrane in liquid interfaces for applications such as transparent windows, filtering, flow sensing, and DNA sequencing.

A better comprehension of the interactions between graphene/liquid interfaces needs to be improved to enhance the design of applied devices. Raman spectroscopy has a significant role in studying graphene and the interactions between graphene and the surrounding medium, such as substrates, gases, and liquids.

Since Raman spectroscopy is sensible to defects, doping, strain, and strain variations, it can access crucial information about the interaction of graphene/liquid interface.

In this work, we present a microfluidic channel with windows covered with suspended graphene to study the interactions of the air/graphene/air (dry) and air/graphene/water (wet) interfaces. Also, we aim to study the dynamics of the optical response of the graphene when we insert water into the channel and let it spontaneously evaporate.

Our results show that the optical response is sensitive to graphene and water''s mechanical and electronic interactions. As a highlight result, we observe the generation of metastable defects in graphene; those defects increase with time when graphene is in contact with water in the channel.

Finally, we transfer an h-BN flake over a graphene window to understand the difference between this window and the free-suspended membrane; we observe that the h-BN flake makes the graphene sample much more stable mechanically and suppresses some of the interaction signatures.

This study''s results attract a deeper understanding of 2D materials'' interactions with liquids.

Plasmon phonon-polaritons in minimally twisted bilayer graphene and talc heterostructures observed by scanning scattering nearfield optical microscopy

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Twisted bilayer graphene (TBG) shows remarkable electronic properties, including superconductivity and correlated insulating states[1]. On electrostatically gated TBG devices with high doping, scattering scanning near field optical microscopy (s-SNOM) reveals triangular domains due to the different optical conductivity of each of the stacking arrangements of the TBG superlattice[2]. In this work, we present a study of plasmon phonon-polaritons in minimally twisted bilayer graphene and talc heterostructures using s-SNOM in the mid-infrared (IR) range without any electrostatic gating. At small twist angles (i.e. angles between 0 and 1.1º), the main part of the triangular domains is constituted by reconstructed regions of stable Bernal (AB or BA) stacking[3], and its boundaries concentrate most of the lattice strain which presents increased optical conductivity[3]. Our experiments were carried out with a mid-IR laser source ranging from 920 cm^{-1}up to 1140 $cm^{-1}$and allowed us to image highly confined plasmon polaritons. In some laser frequencies, we havealso observed sixfold domain wall arrays with the same period as the TBG superlatticewithin the talc reststrahlen band. This work provides new insights into TBG and talc heterostructures electronic and polaritonic properties. We believe our findings will stimulate further research on designing and characterizing novel heterostructures for electronic and optoelectronic applications.

Acknowledgments:
This work was supported by CNPq, INCT/ Nanomateriais de Carbono, CAPES and FAPEMIG, Brazil. The authors are thankful to the Laboratory LCPNano of UFMG.

References:

1. Cao, Y. et al. Unconventional superconductivity in magic-angle graphene superlattices. Nature 556, 43-50 (2018).
2. Jiang, B. Y. et al. Plasmon Reflections by Topological Electronic Boundaries in Bilayer Graphene. Nano Lett. 17, 7080-7085 (2017).
3. Gadelha, A. C. et al. Localization of lattice dynamics in low-angle twisted bilayer graphene. Nature 590, 405-409 (2021).

Room-Temperature Ultrafast Photoluminescence of an Electron-Hole Plasma in 2D TMDs

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The transition metal dichalcogenide (TMD) family of semiconducting 2D materials present remarkable many-body effects such as an exciton Mott transition into electron-hole plasma (EHP) and liquid phases due to their strong Coulomb interactions. However, the light emission in these high carrier density fermionic phases were only investigated at specific conditions that involved suspended samples with large carrier lifetimes or low temperatures. Here, we show that pulsed laser excitation at high pump fluences can generate this EHP phase in as-exfoliated mono and multilayer TMDs ($WSe_2$and $MoSe_2$) at room temperature leading to a large broadening in their photoluminescence [1]. Besides, these high excitation densities induced an indirect-to-direct band gap renormalization in the multilayer WSe_2samples provoking a notable enhancement in their light emission. In agreement with our theoretical calculations, the EHP photoluminescence at high energies displayed an exponential decay that directly reflected the electronic temperature. Moreover, we probed the dynamics of the EHP by two-pulse excitation correlation measurements and we revealed an ultrafast response of less than 100 fs and a slower one of few ps associated with the electron-phonon and phonon-lattice thermalizations, respectively. Our work may shed light on the study of further many body phenomena at high excitation densities in 2D materials and their heterostructures as well as on novel applications of TMDs in optoelectronic devices.

[1] Sousa, F. B. et al. Ultrafast Hot Electron-Hole Plasma Photoluminescence in Two-Dimensional Semiconductors. *Nanoscale*, 2023, **15**, 7154-7163.

Spin-valley physics in strained transition metal dichalcogenides monolayers

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Transition metal dichalcogenides (TMDCs) are ideal candidates to explore the manifestation of spin-valley physics under external stimuli. Here, we investigate the influenceof strain on the spin, orbital angular momenta, and g-factors of monolayer TMDCs within first principles[1]. Our calculations reveal the behavior of direct exciton g-factors under the isolated impact of strain: tensile (compressive) strain increases (decreases) the absolute value of g-factors. Strain variations in the range of ±0.5% modify the bright (A and B) exciton g-factors by 0.3 (0.2) for W (Mo) based compounds and the dark exciton g-factors by 0.5 (0.3) for W (Mo) compounds, suggesting that strain can be responsible for g-factor fluctuations observed experimentally. We complete our analysis for the Gamma and Q valleys, revealing that the spin degree of freedom dominates over the orbital angular momentum. This fundamental microscopic insight into the role of strain in the spin-valley physics of TMDCs provided crucial information to understand recent experiments on strained MoSe2 [2] and WS2 [3] samples.

Acknowledgements:

This research was funded by the DFG SFB 1277 and SPP 2244 projects.

References:

[1] Faria Junior et al., New Journal of Physics 24, 083004 (2022).

[2] Covre, Faria Junior et al., Nanoscale 14, 5758 (2022).

[3] Blundo, Faria Junior et al., Physical Review Letters 129, 067402 (2022).

Stacking order effects on the energetic stability and electronic properties of n-doped graphene/h-BN vander Waals heterostructures on SiC(0001)

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Van der Waals (vdW) systems composed of graphene (G) and hexagonal boron nitride (h-BN) have been extensively investigated, including recent experimental studies on the large-scale synthesis of h-BN on graphene/SiC(0001) templates [2D Materials **5**,025004 (2018)]. Interestingly, their results suggest that besides the vdW epitaxy of h-BN on graphene, confined growth of h-BN monolayers between graphene and SiC(0001) could also take place, leading to h-BN encapsulation. In this work, we have performed a theoretical study, based on first-principles calculations, of G/h-BN heterostructures on SiC(0001), the latter covered by a carbon buffer layer (SiC). Our findings reveal an energetic preference for the encapsulation of h-BN below a single layer of graphene on SiC, viz.: G/h-BN/SiC in the case of bilayer systems and G/h-BN/G/SiC in the case of trilayer systems. Electronic structure calculations show that graphene''s linear energy band dispersion is preserved in the bilayer systems independent of the stacking order, however, the Dirac points lie in different energy positions due to the different electron doping levels of graphene. In the trilayer systems, the n-type doping level of graphene also depends on the stacking order. The electronic band structure of G/h-BN/G/SiC is characterized by two Dirac points, both below the Fermi level, but with different energies, while the other (energetically less stable) systems, G/G/h-BN/ and h-BN/G/G/SiC are characterized by the emergence of parabolic bands near the Fermi level. Additional structural characterizations of these G/h-BN heterostructures on SiC were carried out based on C-1s core-level-shift (CLS) simulations and carbon K-edge X-ray absorption near edge spectroscopy (XANES), with the goal of assisting future experimental spectroscopy in this graphene/h-BN vdW systems.

Structural and optical characterization of some Van der Waals materials: talc, jacutingaite and tilkerodeite

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The possibility to engineer new two-dimensional layered heterostructures to optimize electrical, mechanical, optical and thermal properties of materials of atomic thicknesses opened the necessity to unveil and understand new minerals. Experimental efforts to synthe laboratory analogs of these minerals are valuable due to the fact that in some situations their natural quantities are not sufficient for further characterization and application studies. In this work the materials talc (mineral) [1], jacutingaite and tilkerodeite (synthed)[2,3] are experimentally and theoretically studied by different strategies by using Raman, nano-infrared spectroscopy, and Density Functional Theory. Their structures are understood according to their dimensionalities, with mechanical properties like their lubricity being explored and compared to other known good lubricants like graphite. The phonon modes are studied, being the results fundamental to understand their applications, and on how to engineer their properties with controlled modifications. Acknowledgements: Fapemig, CNPq, INCT Nanocarbono, Rede 2D Fapemig, Sdumont supercomputer, CENAPAD-SP, CENAPAD-RJ/LNCC. References: [1] Longuinhos, R., et al. J. Phys. Chem. C 127.12, 5876-5885, (2023). [2] Longuinhos, R. et al. J. Phys.: Condens. Matter 33, 065401 (2021). [3] Longuinhos, R., et al. J. Raman Spectrosc 51.2, 357-365 (2020).

Study of Defects as a function of the temperature of synthesis and He ions bombardment doses of MoSe2 by CVD

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In the last decade, transition metal dichalcogenides (TMDs) have been intensively synthesized and studied, allowing a direct correlation of their morphological and physical properties. Nevertheless, the full potential for applications of TMDs intrinsically depends ona deeper understanding of defect type and distribution in these materials. Quantifying and identifying defects in TMDs is therefore mandatory to reach state-of-the-art prototypes and functional optoelectronic devices. In this work we synthesize MoSe2 by APCVD using three different temperatures. The resulting samples are then investigated by Raman and photoluminescence (791nm to 816nm, when we increase the synthesis temperature) spectroscopy as well as by Atomic Force Microscopy. Finally, MOSFET devices are made in order to allow a precise measurement of the electrical resistance as a function of the growth temperature. The relation of Raman spectra and defect density1 and synthesis temperature is established, and a comprehensive comparison with electrical conductivity of MoSe2 carried out. In another work, we are studying the PL as a function of He ions bombardment doses and temperature in MoSe2 monolayers. Here we note a decrease in the intensity of the PL with increasing doses, in addition to a redshift and an increase in the intensity of the spectra when we decrease the temperature. These works sheds light into methods for macroscopic quantification of defect density in TMDs, a crucial parameter to tailor technological applications.

Study of glyphosate adsorption on graphene-based substrates: a DFT study

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We face a period in history when half of the world's population faces water scarcity, and soon the second half of the world's population will face water scarcity also - and climate changes are accelerating extreme weather events, such as long periods of drought. The misuse of herbicides worldwide, headed by glyphosate (GLY), causes fauna, flora, and human health problems. Therefore, studies aimed for the GLY detection and/or removal from aquatic environments are of great importance for the future of mankind. Graphene-based nanomaterials are one of the most promising new technologies to remove GLY and other emergent surfactants. In this direction, we combined a potential energy surface exploration, via *ab initio*molecular dynamics simulations in six different graphene-based chemical environments, from pristine to cased with point defects: monovacancy, doping (nitrogen or oxygen), and adatoms (nitrogen or oxygen), with high-level quantum mechanical optimizations based on density functional theory within PBE Exchange-correlation functional with van der Waals corrections and the projector augmented wave method. Our analysis revealed a rich GLY reaction environment, ranging from physisorption in the case of pristine, N-, and O-doped graphene, with adsorption energy intensities (E_{ad}) lower than 1 eV, with large substrate-molecule distances and total magnetic moments null; up to chemisorption in the case of graphene with monovacancy ($E_{ad} \sim 2.5$ eV), presenting a high interaction energy and total magnetic moments around 2 μ_B, bringing typical characteristics of substrate poisoning. We highlight the GLY interaction with N and O adatom substrates, where the GLY molecule picks up the adatom from the substrate, givingrise to different molecular compositions. In short, our work intends to provide an atomic-level picture of the interactions that can govern the carbon-based devices applied in the glyphosate detection/removal.

Substrate-Induced Changes on the Optical Propertiesof Monolayer WS2

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Among the most studied semiconducting transition metal dichalcogenides (TMDCs), WS_2showed several advantages in comparison to their counterparts, such as a higher quantum yield, which is an important feature for quantum emission and lasing purposes. We studied transferredmonolayers of WS_2on a drilled Si_3N_4substrate in order to have insights about on how such heterostructure behaves from the Raman and photoluminescence (PL) measurements point of view. Our experimental findings showed that the Si_3N_4substrate influences the optical properties of single-layer WS_2. Beyond that, seeking to shed light on the causes of the PL quenching observed experimentally, we developed density functional theory (DFT) based calculations to study the thermodynamic stability of the heterojunction through quantum molecular dynamics (QMD) simulations as well as the electronic alignment of the energy levels in both materials. Our analysis showed that along with strain, a charge transfer mechanism plays an important role for the PL decrease.

Super-strong optical phonon energy renormalization measured by spatially coherent tip-enhanced Raman spectroscopy in a graphene device

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Optical phonon coherence length (Lc) and group velocity (vg) of graphene as a function of Fermi energy are obtained by spatially coherent tip-enhanced Raman spectroscopy (TERS). It is observed that Lc decreases when moving the Fermi energy close to the neutrality point, which is consistent with the concept of the Kohn anomaly within a ballistic transport regime. An unusually large variation of vg, reaching twice the value for the longitudinal acoustic phonon, is observed for the longitudinal optical phonon close to the Brillouin zone center (Γ point). The strength of this phonon energy renormalization, as observed here, might not be observed by any other technique due to the lack of resolution to measure the phonon dispersion near the center of the Brillouin zone and it is much higher than what has been reported in the literature [1].

Acknowledgments: CAPES.

References:[1] Maultzsch, J.; Reich, S.; Thomsen, C.; Requardt, H.; Ordejón, P. Phonon dispersion in graphite. Physical review letters 2004, 92, 075501

Understanding the oxidation of redox exfoliated NbS2

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Two-dimensional (2D) materials have been extensively explored for the development of new and sustainable devices. Among other applications, their high surface-to-volume ratio leads to a promising range of catalytic processes. In the family of 2D materials, transition metal dichalcogenides (TMDs), are a class of layered materials with structure MX2 (M = transition metal and X = chalcogen). In this context, niobium disulfide (NbS2) is a less explored TMD with a metallic character. Although it is a superconductor at low-temperatures[1] and demonstrates great potential to be applied as catalyst or co-catalyst for hydrogen evolution reaction[2], only few reports demonstrated the synthesis of few-layer NbS2. However, before applications can be pursued, it is essential to understand the main characteristics of the obtained material and its stability under atmospheric environment. In this work, we conducted a thorough characterization of redox exfoliated NbS2 nanoflakes, relating our findings to their structure and stability in oxygen-rich environment.[3] Structural, morphological and spectroscopic characterization demonstrated different fingerprints associated with distinct oxidation stages and processes. This led us to identify oxide-species and analyse the stability of the redox exfoliated NbS2 nanosheets in air, suggesting the most likely reaction pathways during the NbS2 interaction with oxygen, which agrees with our density-functional theory results. Understanding the stability of layered materials is of paramount importance to target future applications, mainly because the electronic properties of these materials strongly depend on their integrity.

References:

[1] C. Witteveen et al. Dalton Transactions, vol. 50, no. 9, pp. 3216-3223, 2021.
[2] J. Yang et al. Nat Mater, vol. 18, no. 12, pp. 1309-1314, 2019.
[3] D. A. Nagaoka et al. Physical Chemistry Chemical Physics, vol. 20, pp. 9559-9568, 2023.

Water-in-water Pickering emulsions stabilized by talcor kaolinite layered nanoparticles.

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Kaolinite - $Al_2Si_2O_5(OH)_4$ and talc - $Mg_3Si_4O_{10}(OH)_2$ are abundant clay minerals which present layered particles with nanometric thickness and few micrometers in length. Such characteristics make them materials of interest in the stabilization of emulsions with low interfacial tension like in water-in-water emulsions. After characterization of the layered particles by several instrumental techniques, the effect of adding layered particles on water-in-water emulsions formed by aqueous colloidal dispersions of amylopectin (AMP) and xyloglucan (XG) polymers was evaluated macroscopically, by confocal microscopy and by measurements of the apparent contact angle. Due to a similar affinity for both liquids and their relatively larger size, talc particles presented the Pickering effect [1], positioning themselves in the droplet interfacial region. Despite the smaller size of kaolinite particles and their greater wettability by AMP phase than XG one, the same effect was also observed. In both cases, it was not sufficient to stabilize emulsions for long periods, since the difference of densities between the particles and the aqueous phases led to particle sedimentation even when adhered to the droplets, generating a higher particle concentration at the bottom of the droplets and an incomplete coating of the interface. To avoid this effect, emulsions with a third element that promotes the system gelation might be prepared [2].Acknowledgements:This study was financed by CAPES - Finance Code 001, CNPq (FW: 300988/2019-2; RAF: 301172/2016-1; 430451/2018-0; 303312/2019-0) and FINEP. ARS thanks CNPq for the Postdoctoral fellowship (163817/2020-0), VVSM and BA thank CAPES for the PhD and Ms.Sc. scholarship, respectively.References:[1] B. Hazt, H.P. Bassani, J.P.E. Machado, J.L.A. Buzzo, J.L.M. Silveira, R.A. de Freitas, Food Hydrocolloids, 104 (2020)[2] C. Pires, B. M. Régnier, M. J. R. dos Santos, R.A. de Freitas, Food Hydrocolloids, 141 (2023)

A comprehensive study of degassing effects on BET surface area of graphene oxide

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Graphene oxide (GO) is an amorphous compound consisted in an oxygen groups-containing 2D honeycomb-like carbon structure, of general (non-stoichiometric) composition $C_xH_yO_z$(y= 0.8 and 1.5 to 2.5 C/O ratio). Due to its great mechanical properties, such as large theoretical specific surface area, GO is regarded as promising material for several applications like energy storage and multifunctional composites [1-3]. In order to determine the surface area of the GO sheets by adsorption of a gas using BET method, the degassing process is always accompanied by the heating of the sample. Although graphene and related materials (i.e., GO, rGO) have been studied over the last 40 years, as far as the authors are aware, there is no reports about the degassing effects on the GO structure and surface area. In this work, the influence of the degassing temperature, sample mass and pre-drying process on the surface area of GO were investigated. The GO sample was submitted to degassing at different temperatures: 50 °C, 80 °C, 150 °C, 250 °C and 350 °C (pre-dried andnot pre-dried). The analyzed mass varied in 100 mg, 150 mg and 200 mg. As result, theadsorption measurement was optimized with degassing temperature of 50 °C, sample massof 200 mg and the pre-drying process decreases the degassing time, but this is not a crucialstep to determine BET surface area. In conclusion, the relation between reduction degree vs. degassing temperature vs. surface area could be established.

Acknowledgments: CNPq, FAPERJ and Finep.

[1] P. P. Brisebois and M. Siaj. Harvesting graphene oxide-years 1859 to 2019: a review of its structure, synthesis, properties and exfoliation. J. Mater. Chem. C. v. 8 p. 1517-1547. 2020

[2] Y. Zhu, et al. Graphene and Graphene Oxide: Synthesis, Properties, and Applications. Adv. Mater. v. 22. p. 3906-3924. 2010

[3] S. Pan and I. A. Aksay. Factors Controlling the Size of Graphene Oxide Sheets Produced via the Graphite Oxide Route. ACS Nano. v. 5. p. 4073-4083. 2011

A molecular dynamics calculation of the structural properties of functionalized graphene nanoscrolls

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Nanoscrolls are nanosized membranes rolled into a cylindrical shape with a spiral side-view, where this membrane can be any 2D material [1]. In this work we apply molecular dynamics simulations, using reactive and classical force fields [2], to study the structural and thermal stability (up to 1000 K) of nanoscrolls made out of functionalized graphene sheets. The functionalization of the graphene sheet is made with oxygen-based functional groups [3], and we study the relevance of the ratio between carbon and functionalized groups have in the formation and the stability of the nanoscroll. This was elucidated through the calculation of the energy profile of the formation process of the nanoscroll. Our results demonstrate that the nanoscrolls of functionalized graphene are stable at the simulated temperatures.

References:

[1] S. Braga et al., Nano Letters 4, 881-884 (2004)
[2] K. Chenoweth et al., J. Phys. Chem. A 112, 1040-1053 (2008)
[3] P. Mazon and C. Woellner, (submitted)

An efficient MATLAB code to predict effective properties of composite materials using the finite-volume theory

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This paper presents an efficient computational tool developed in MATLAB to evaluate the effective elastic properties of composite materials with periodic microstructures. The basic building block can characterize the microstructure periodicity called a repeating unit cell of 2D periodic material [1]. The effective elastic properties are determined using a numerical homogenization approach based on the Finite-Volume Theory analysis ([2] and [3]). These effective elastic properties are affected by the constituents' architectural arrangements, volume fractions, and stiffness contrast. The computational code can be easily applied for composite with N-phase, including porous material. Numerical examples involving materials with periodic microstructures are analyzed. The results demonstrate the Finite-Volume Theory formulation's performance for evaluating homogenized or effective elastic properties by comparing with results obtained by the finite-element method [4] and analytical solutions presented in the literature.

References:

[1] A. S. Drago, M.-J. Pindera, “Micro-macro mechanical analysis of heterogeneous materials: macroscopically homogeneous vs periodic microstructures”. Composites Science and Technology, vol. 67, n. 6, pp. 1243-1263, 2007.

[2] Y. Bansal, M.-J. Pindera, “Efficient reformulation of the thermoelastic higher-order theory for functionally graded materials”. Journal of Thermal Stress. vol. 26, n. 11-12, pp. 1055-1092, 2003.

[3] M. A. A. Cavalcante, M.-J. Pindera, H. Khatam, “Finite-volume micromechanics of periodic materials: past, present, and future”, Composites, vol. 43, pp. 2521-2543, 2012.

[4] E. Andreassen, C. S. Andreasen, “How to determine composite material properties using numerical homogenization”. Computational Materials Science, vol. 83, pp. 488-495, 2014.

Anisotropic Hydrogenation Pattern in a new 2D Carbon Allotrope

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The discovery of graphene has renewed interest in 2D carbon nanostructures. Specifically, researchers have proposed new structures by combining hexagonal and other polygonal carbon rings. Recently, Bhattacharya and Jana introduced a new 2D carbon allotrope called Tetra-Penta-Deca-Hexagonal graphene (TPDH-Gr), composed of rings with 4, 5, 10, and 6 carbon atoms [1]. This unique topology results in distinct mechanical, electronic, and optical properties, including potential applications in UV protection. As with other 2D carbon allotropes, TPDH-Gr''s physical and chemical properties can be modified through chemical functionalization. However, the hydrogenation dynamics of TPDH-Gr have yet to be explored, even though hydrogenation is a typical method for functionalization typically occurring in experimental synthesis. In this work, we investigated the hydrogenation dynamics of TPDH-Gr and its effects on the electronic structure using DFT and fully atomistic reactive Molecular Dynamics simulations. This methodology has been successfully used in previous research [2]. Our findings indicate that H atoms are mainly incorporated on tetragonal ring sites, resulting in well-defined pentagonal carbon stripes. The hydrogenated structures show narrow band gaps and the presence of Dirac cones, indicating anisotropic transport properties.

Acknowledgments:

The authors thank PRH-ANP (PRH.49-UFABC) and the Brazilian agency CNPq (process #310045/2019-3) for the financial support and CCM-UFABC for the computational resources provided.

References:

[1] D. Bhattacharya and D. Jana, Physica E: Low-Dimensional Systems and Nanostructures, 127, 114569, 2021.

[2] E. Marinho and P. A. da Silva Autreto, Physical Chemistry Chemical Physics, 23, 9483-9491, 2021.

Bioprinting 3D GelMA/graphene scaffolds at low temperature for tissue engineering applications

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3D bioprinting is a promising technique for biomedical applications. When aligned to low temperature, it promotes the development of biomimetic scaffolds for applications in tissue engineering, providing high resolution to the structure and improved mechanical properties [1]. However, in the current context, there are difficulties about the selection of the applied bioinks as well as limitations when the infusion of cells and consequently the construction of tissues [2]. In the present study, GelMA with graphene at different concentrations were synthed at room temperature and at low temperature for biomedical applications to analyze the mechanical properties derived from graphene and when subjected. GelMA was synthed according to the protocol [3]. Subsequently, a solution of GelMA was prepared at different concentrations of graphene. The scaffolds were printed in 3D using an Allevi 2 Bioprinter. (Descrever um pouco dos resultados). The samples were then characterized morphologically, mechanically, and structurally. The results showed that the structures obtained from the GelMA/Grafeno hydrogels produced have potential for applications in tissue engineering.

Controlling Crystal Phase Transition of MoS2 with ion beams

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Recently, transition metal dichalcogenides, or simply TMDs, have attracted great attention due to their vast application field [1]. Among these materials, molybdenum disulfide (MoS2),a two-dimensional material with interesting electronic and optical properties, stands out. However, its properties are significantly influenced by both the number of layers formed and its crystalline structure, which can be of two main forms: 2H and 1T [2]. The 2H crystalline form is the most common and stable form of MoS2, being a direct bandgap semiconductor frequently studied for use in electronic devices. The 1T crystalline form of MoS2 presents metallic properties with high electrical conductivity. Due to the importance of controlling such properties and to apply them adequately, investigation of the induction of phase transition control is crucial. Considering all these aspects, the present work investigates the crystal phase transition of MoS2 induced by ion bombardment. Energetic He ions are used to remove sulfur atoms, leading to phase transformation of the crystalline structure. Modifications are tracked by various techniques, such as X-Ray Induced Photoelectron Spectroscopy (XPS), Raman spectroscopy, and Photoluminescence (PL). Results obtained so far have evidenced that phase modification can be induced by ion beams, provided a careful choice of irradiation time and ion species.

Acknowledgements:
The authors thank INCT-Namitec, CNPq, CAPES, FAPERGS, and LASIS for the financial support.

References: [1] Chhowalla, M., et al., The chemistry of two-dimensional layered transition metal dichalcogenide nanosheets. Nature Chemistry vol 5, page 263-275 (2013). [2] Zhu, J., et al., Argon Plasma Induced Phase Transition in Monolayer MoS2. Journal of the American Chemical Society vol 139, page 10216-10219 (2017).

Development of 2D materials based on SnS_2 doped with RE^{3+} ions for application in photonics

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2D materials have been explored due to their photocatalytic properties by reason of their physical and chemical properties. Tin derivatives sulfides can be obtained in different phases as SnS, SnS_2, Sn_2S_3, Sn_3S_4, and Sn_4S_5[1]. $SnS_2$is an n-type semiconductor with hexagonal structure with applications in nanoelectronics, optoelectronics, catalysis and sensors. The introduction of the rare earth ions (RE^{3+}) into the lattice can modify their features and functionality, changing the semiconductor from n-type to p-type [2]. This work reports a hydrothermal synthesis at 180 ºC for 6h of SnS_2materials with and without RE^{3+}, starting from SnCl4.5H2O and thiourea as precursors. From the X-ray diffractogram, the SnS2 phase with a hexagonal structure was identified, with a space group P63mc (JCPDS no. 43003) and cell parameters set as a=53.645 b=3.645 c=11.802. Luminescence and photocatalytic properties are in progress and will be explored to evaluate their potentiality to be applied in photonics as photocatalytic system driven by upconversion and NIR sensors.

Acknowledgments:

We acknowledge FAPESP, CAPES and Cnpq.

References

[1] H. Zhang, Y. Balaji, A. N. Mehta, M. Heyns, M. Caymax, I. Radu, W. Vandervorstb and A.Delabie. Formation Mechanism of 2D SnS2 and SnS by Chemical Vapor Deposition using SnCl4 and H2S. Journal of Materials Chemistry, vol. 6, p. 6172-6178 (2018).

[2] G. Bai , S. Yuan , Y. Zhao , Z. Yang , S. Y. Choi , Y. Chai , S. F. Yu , S. P. Lau , and J. Hao. 2D Layered Materials of Rare-Earth Er-Doped MoS 2 with NIR-to-NIR Down- and Up-Conversion Photoluminescence. Adv. Mater., vol. 28, p. 7472-7477 (2018)

Development of a Graphene Oxide-Coated Long-Period Fiber Grating Sensor for Carbon Dioxide Detection

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Currently, the detection of Carbon Dioxide (CO2) has been a factor of extensive investigation, due to the various problems that this gas can cause to the environment, the health of humans and animals, and the agri-food industry [1]. In this context, the objective of this work is to develop CO2 sensors based on optical fibers of the LPG (Long Period Grating) coated with graphene oxide (GO). The sensors with optical fibers have certain advantages compared to other sensors, such as immunity to electromagnetic interference, compact size, multiplexing capability, easy integration to remote sensing, and high corrosion resistance. LPGs are based on diffraction gratings, i.e., a periodic modulation in the refractive index along with the core of the optical fiber, which is a technique widely used to improve the detection efficiency of optical fibers. LPGs do not have a high sensitivity to CO2 thus, it is necessary to use some coating on their grating to increase their sensitivity for gas detection. Thus, by coating the core of these fibers with GO, it is possible to improve their detection system. Recent studies show that GO has a significantly shorter response time compared to other materials for CO2 detection, at various concentrations and under varying humidity and temperature conditions [2]. One of the factors that contribute to this is the high surface area that this nanomaterial has, which allows a greater biomolecular interaction per unit area and can contribute to the immobilization of gas molecules. We are currently performing preliminary tests with GO-coated LPG for the sensing of various CO2 concentrations.

Acknowledgments:The authors thank CAPES, CNPq, and Fapemig for funding this work.

References: [1] Zhu, J. et al. Chinese Chem. Lett. 29, 1445-1450 (2018).

[2] Akhter, F., Alahi, M. E. E., Siddiquei, H. R., Gooneratne, C. P. Mukhopadhyay, S. C. IEEE Sens. J. 21, 4241-4249 (2021).

Development of Hybrid Microfibers Based on Graphene Derivatives and Carbon Nanotubes

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Since graphene was discovered in 2004, has attracted the attention of many researchers, due to its outstanding electrical, thermal, and optical properties. Based on that, graphene and its derivatives, have been touted as a candidate for the development of microfibers, due to their superior properties concerning conventional carbon fibers [1]. Previously, we reported the development of graphene derivative microfibers with controlled and homogeneous shapes and tunable diameters, fabricated exploring the 3 dimensional (3D) hydrodynamic focusing concept on a microfluidic device [2]. However, there is still room for improving the mechanical properties of neat graphene derivatives microfibers. One interesting approach consists of the use of molecular-like materials that can act as a bridge between the GO nanosheets, improving the binding between them. In this way, we address an optimization study of the incorporation of carbon nanotubes (CNTs) on GO dispersion, aiming for the preparation of graphene derivative fibers with superior mechanical properties. The GO sheets were obtained by modified Hummer´s method [3] and characterized by Raman spectroscopy. We investigate the effect of mild oxidation of single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) to obtain stable aqueous dispersion. For this, we performed the procedure described by Rosca et al [4], followed by the sonication in ultrasound tip and ultrasound bath at a frequency of 24 Hz and power of 280 W in 0.5 cycles for one hour and twenty minutes. It was possible to obtain stable CNTs dispersion by the low insertion of oxygen functional groups. We evaluated the effect of the incorporation of the aqueous dispersion of 1 mg/mL SWCNTs ox. and MWCNTs ox. in the GO dispersion (5 mg/mL) pointing to the preparation of GO microfibers, using a CTAB 0,05%. We obtained GO fibers longer than 5 cm. We demonstrated a systematic study of the fabrication of hybrid CNTs ox - GO fibers by wet-spinning.

Development of Inexpensive Laser-Induced Graphene Sensors for Electrochemical Detection of NADH

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The use of graphene-based materials in electrochemical sensing has gained significant interest due to their unique properties, such as high surface area, conductivity and electrocatalytic behavior [1]. Among these materials, Laser-Induced Graphene (LIG) has become increasingly popular. LIG can be fabricated on flexible substrates using a fast and simple laser irradiation process, making it an attractive option for electrochemical sensor development. In this study, a low-cost electrochemical sensor was fabricated using LIG for the detection of nicotinamide adenine dinucleotide (NADH) at a low potential of 50 mV vs Ag/AgCl. The LIG was produced using mild laser power conditions (1.5 W laser power at 405 nm) on a polyimide substrate, resulting in electrically conductive graphene with a sheet resistance of 24.38 ± 2.19 Ω/□ and a porous 3D morphology rich in defects. Pseudo-reference electrodes based on Ag/AgCl were obtained by a simple chlorination approach on a deposited silver layer, resulting in a stable potential with a potential difference of less than 2.5 mV vs Ag/AgCl (3 mol L^{-1}KCl) commercial electrode. The electrochemical LIG-based sensors were then used to detect NADH, an essential coenzyme for neurotransmitter activities, with a linear range of 0.05-10 mmol L^{-1}. The presence of residual oxygenated functional groups (C-O/C=O) on the LIG structure was found to be essential for the electrocatalytic activity towards the electrooxidation of NADH at low potential [2]. Overall, the electrochemical LIG-based sensors show promise in the development of biosensors for the sensitive detection of pathogens, such as bacteria.

Acknowledgements:

MackPesquisa, FAPESP, CNPq, CAPES, FINEP and INCT NanoVida.

References:

[1] Zhao, L. et al. ACS Appl. Mater. Interfaces (2023) doi:10.1021/acsami.2c20859

[2] de Camargo, M. N. L. et al. Eletrochim. Acta 197, (2016) 194-199.

Direct Synthesis of CeO2:SBA-15 Nanocomposites inIonic Liquid Medium for Enhanced Catalytic Applications

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Cerium oxide-based nanocomposites are an attractive option for use in environmental catalysis applications due to their unique structural, textural, and morphological properties. To create promising materials for use in this field, researchers have investigated the development of nanocomposites containing CeO2 and SBA-15 as a support for catalytic metals (CM), including both noble (Pd, Pt and Rh) and non-noble (Ni and Co) metal-based catalysts. The incorporation of CeO2 enhances the catalytic activity by promoting the dispersion of CM in the mesoporous structure of SBA-15, thus avoiding agglomeration, and providing better interaction between the metal-support. Additionally, SBA-15 increases the thermal stability of the CM, preventing sintering. To prepare such support materials, CeO2 precursor salt (Ce(NO3)3.6H2O) was incorporated in SBA-15 using the direct synthesis method. Imidazolium-based ionic liquids (ILs) with different alkyl chains and anions (Br- and BF4-) were employed as a reaction medium to study the effect the salts in the preparation of the mentioned nanocomposites. The SAXS curves revealed that all nanocomposites have a 2D hexagonal mesostructured characteristic of SBA-15. The DRX analysis confirmed the crystalline cubic fluorite-type of space group Fm3m structure of CeO2, as also observed by Raman spectroscopy. The surface area (BET), was found to be lower for the nanocomposites prepared with the IL with BF4- anion. However, these nanocomposites had a higher average pore diameter when compared to the ones prepared with Br- anion. SEM images were used to visualize the morphology and particle size of the material, which was found to be influenced by the used IL. The researchers comparatively studied the structural, textural, and morphological properties of the materials, and found that the IL anion hydrophobicity influences the mesophase, leading to larger pores.

Acknowledgments:
CAPES, INCT/INEO, FAPESP for the financial support.

Electrochemically reduced graphene oxide onto flexible interdigitated electrodes as strain sensors

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Graphene oxide (GO) is a graphene derivative obtained cheaply by the Hummers'' method. It is an electrical insulator that becomes a good conductor when subjected to a reduction treatment (usually chemical or thermal). This study presents the electrochemical reduction of GO to rGO (reduced graphene oxide) directly onto flexible interdigitated gold electrodes and its application as strain sensors. The ultimate goal is the development of sensors using electrochemical reduction alternatively to thermal and chemical reduction routes, which use dangerous reagents or a large amount of energy. Therefore, the GO reduction was performed using cyclic voltammetry (-1.5 V to 0 V and 0 to -1.5 V), and it was found that only one cycle is necessary to reduce the GO to rGO with low resistance (<100 Ω). A typical graphene-like behavior was confirmed when the devices were applied as electrolyte-gated field-effect transistors. As strain sensors, rGO onto flexible interdigitated gold electrodes presented a GF (gauge factor) average of 3.4, a value ca. 60 % higher than conventional metallic strain sensors. Acknowledgement Fapesp process 18/07508-3.

Electronic properties of MoS_2 modified by Ionic Liquid functionalization

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The unmatchable properties of two-dimensional (2D) layered transition-metal dichalcogenides (TMDs) have been drawing appreciable attention. Varying TMD's composition allows to tune band gaps between 0 to 2 eV. Such versatility allows their use as metal or semiconductor materials. Furthermore, monolayer TMD'S exhibit a direct band gap allowing their use in optoelectronic devices. In order to take advantage of all these properties, and enhance some features, functionalization of different sources are vastly studied. Photoluminescense (PL) enhacement or quenching is intrinsically related not only to material layer's numbers, as also to the functionalizing molecules. PL also shows different responses depending on the solvent used on solution preparation. In our investigation, $MoS_2$was synthed by sulfurization of sodium molybdate. An aqueous solution of this salt was spin coated on the surface of a silicon substrate. Following the sulfurization step, $MoS_2$was obtained in most of the sample surface. Functionalization with two distinct ionic liquids were performed by drop deposition. We used 3-butyl-1-methyl-1Himidazol- 3-ium bis((trifluoromethyl)sulfonyl)amide which is highly hydrophobic and 1-Butyl-3-methylimidazolium hexafluorophosphate both diluted in dichloromethane. XPS, Raman, and PL results indicate functionalization of MoS_2in addition to modification of its electronic properties seen by differences in PL results.

Acknowledgements:

We would like to thank the financial support of INCT INES, MCTIC/CNPq, CAPES, and FAPERGS.

References: [1] Liu, H.,Gi, g., et al. ACS Applied Materials Interfaces,12, 13174–13181 (2020). [2] Gao,J.,Li, B., et al. ACS Nano,10, 2628–2635 (2016). [3] Mouri, S., Miyauchi, Y., et al. Nano Letters, 13 (12), 5944-5948 (2013).

Evaluation of electrical and electromagnetic propertiesin PC/ABS blends-based graphene nanoplatelets and carbon nanotubes nanocomposites

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Polycarbonate (PC)/acrylonitrile-butadyene-styrene copolymer (ABS) blends are commonly applied in portable electronics. For this application, some polymer parts need addition of metallic components, or a posterior metallization process, to promote electromagnetic compatibility (EMC) of the electronic enclosures. An alternative way to improve the EMC in PC/ABS blends is the addition of conductive fillers as graphene nanoplatelets (GNP) and multi-wall carbon nanotubes (MWCNT) [1-2]. In this work, PC/ABS blends (85/15) with the addition of 3 wt% of GNP (MGgrafeno, Brazil) and 1.5 and 2 wt% of MWCNT (NC7000, Belgium) were prepared in a twin screw extruder (AX Plásticos, model AX16:40DR). Specimens were prepared by injection molding (Battenfeld, model Plus 350/75). The nanocomposites were characterized by Field Emission Gun Scanning Electron Microscopy (FEG-SEM, TESCAN - MIRA3), impedance spectroscopy (Impedance/Gain-phase Analyzer, model Solartron SI 1260) and the electromagnetic interference shielding (EMI SE) in the X-band (8.2-12.4 GHz) were measured in a vector network analyzer (VNA, Agilent Technologies, model PNA-L N5235A) with a coupled waveguide (WR-90). The injection molding process interferes in the formation of the morphology, since it can favor the orientation of the GNP in the direction of the molding flow, hindering or even destroying the ideal three-dimensional formation. The addition of 3 wt% of GNP promotes a total attenuation (SET) of 5 dB. The addition of 1.5 or 2 wt% of MWCNT in the PC/ABS/GNP composition promote a sinergistic effect with the increase the SET values for 19-20 dB. These hybrid nanocomposites are a promising material for future EMI SE applications with a more substantial attenuation.

Acknowledgements:
The authors thank CNPq (Process 440132/2021-3) for financial support.

References:
[1] S. Maiti and B.B. Khatua Polymer Composite, 37, 2058 (2016).
[2] E.G.R. Anjos et al. Journal of Applied Polymer Science, 139,51946 (2022).

Evaluation of photothermal properties of size-dependentreduced graphene oxide

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Graphene is a two-dimensional material formed by carbon atoms in a hexagonal lattice. This carbon allotrope is known for its interesting properties, such as mechanical strength and high electrical and thermal conductivity [1]. Moreover, graphene and its derivates is also exploited for biomedical applications due to their ability of absorbing NIR light and transforming it into local heat. Normally, graphene sheets have micrometric scale, which can be a limiting factor on cellular internalization. Hence, in this work, we studied the effect of the photothermal activity of several sizes of reduced graphene oxide sheets aiming a better proportion between size and its ability on converting NIR radiation. For that matter, graphite flakes were oxidized following a procedure described in literature, producing graphite oxide (GO) [2]. Thus, six GO dispersions were prepared and exfoliated at different times, using either an ultrasonic bath, at frequency of 40 kHz (2 h, 4 h and 6 h) or a probe sonication, at a frequency of 20 kHz (30 min, 1 h and 2 h) [3]. The resulting dispersions were then lyophilized. The following steps include i) Raman spectroscopy characterizations, in order to evaluate defects degrees after the exfoliations processe ii) scanning electronic microscopy (SEM) to determinate a size distribution and iii) evaluate the photothermal activity ofthe chemical reduced form of GO (rGO) by irradiating the materials with a red laser, varying the dispersion concentration.

Acknowledgments:

UFF, CAPES, CNPq, FAPERJ, PPGQ-UFF, LAME, LAMATE

References:

[1] K. L. S. Castro et al. Int. J. Electrochem. Sci., 13, 71 - 87 (2018)

[2] T. C. dos Santos et al. J. CO2 Utilization, 48, 101517 - 101528 (2021)

[3] C. Mellado et al. Materials Today Chemistry 13, 1 - 7 (2019)

Exciton dynamics in hBN/WSe2/hBN heterostructures induced by surfaceacoustic waves

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In 2D materials-based heterostructures, the dynamic strain and piezoelectric potential modulation carried by the SAW strongly interact with optically generated electron-hole pairs. In this contribution, we investigate the acoustically modulated charge and exciton dynamics in WSe2 monolayers (1L) encapsulated in hexagonal boron nitride (hBN) layers onto LiNbO3 substrates. Below 200K, we show that the exciton dynamics can be efficiently modulated by the SAW with very fast on and off times. Quenching of the photoluminescence (PL) emission, which can be controlled with the acoustic power, is attributed to the effective interaction of the excitons and the SAW piezoelectric field [1,2]. We also generate standing wave patterns on the sample surface, demonstrating carrier localization in the wave antinodes. At room temperature (295K), the exciton dynamics under acoustic waves is clearly affected by the presence of extra charge carriers introduced in the overall dynamics. These carriers may be optically injected from the substrate [3] or from traps associated with defects or potential fluctuations in the hBN/1L-WSe2/hBN heterostructure. As the SAW power is increased, the extra carrier density leads to an initial gain in photoluminescence emission, due to screening of the SAW piezoelectric field, followed by an overall PL quenching at higher acoustic powers. Therefore, we observe two different carrier transport regimes which may help us to find more effective means to manipulate fundamental excitations in 2D transition metal dichalcogenides systems.

Acknowledgments: The authors are grateful to FAPESP (2021/06803-4), CAPES (88887.372038/2019-00) and CNPQ (306201/2022-4) for financial support. LNnano/CNPEM and LAMULT-IFGW for experimental support

[1] K. Datta, Z. Li, Z. Lyu, and P. B. Deotare, ACS Nano 15, 12334-12341 (2021).
[2] D. Scolfaro et al., ACS Nano 15, 15371-15380 (2021).
[3] V. O. Gordo et al., Nanoscale 10, 4804-4815 (2018).

Experimental Design of MoS2-GO Heterostructures for Hydrogen Catalysis

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The search for a more sustainable energy matrix has been an urgent topic in science, and the use of Hydrogen presents itself as an interesting alternative. The challenge of implementing this technology lies in the lack of a stable catalyst, with high catalytic density and composed of materials abundant on Earth. In this context, two-dimensional materials, such as Molybdenum Disulfide (MoS2) and its heterostructures, may present catalytic activity for the Hydrogen Evolution Reaction (HER). However, this activity is concentratedat the edges and defects of the material.

To modify these characteristics, it is possible to add a conductor material or a dopant that activates the basal plane of MoS2, increasing its conductivity and catalytic activity. We used Graphene Oxide (GO) to build a heterostructure where GO and MoS2 films are stacked. To produce this heterostructure, a 2^3 factorial design was used, which consists of a variation of synthesis conditions such as temperature, compound concentration, and synthesis time.

Using Ammonium Tetrathiomolybdate ((NH4)2MoS4) precursor, which was diluted in water and mixed with GO solution and deposited on SiO2 substrates, it was taken to the oven for thermal decomposition of the reagents. The results were evaluated through Optical Microscopy, Atomic Force Microscopy (AFM), and Raman Spectroscopy to analyze the surface of the samples and layer composition.

Acknowledgements: We would like to thank Serapilheira for the scholarship.

References: [1]Yu Lei,et al.Low-temperature Synthesis of Heterostructures of Transition Metal Dichalcogenide Alloys (WxMo1-xS2) and Graphene with Superior Catalytic Performance for Hydrogen Evolution.ACS Nano,11,5, 5103-5112.2017.

GRAPHENE OXIDE NANOFLUIDS FOR ENHANCED OIL RECOVERY (EOR)

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Chemical methods used for enhanced oil recovery (EOR) are based on reducing interfacial tension, improving sweeping efficiency, and reversing rock wettability to increase oil displacement in the reservoir. Graphene oxide (GO) based nanofluids have the potential to be effective EOR additives due to their ability to affect the physicochemical properties of fluid-fluid and rock-fluid systems. The oxygenated groups on the surface of GO provide stability in aqueous medium and potential sites for chemical modification to improve stability and effectiveness. While surfactant injection is commonly used in EOR, it can present problems such as loss by adsorption and instability under adverse salinity and pressure conditions. In this study, we evaluate the use of GO in different degrees of oxidation as a carrier for surfactant nanofluids in EOR. The GO is characterized by SEM and AFM, FTIR, and Raman to determine its structural and morphological properties. Zeta potential measurements are used to assess the colloidal stability of GO under various conditions. Wettability is evaluated by measuring the contact angle of calcite fragments aged in crude oil and fragments treated with the nanofluid. The performance of GO-based nanofluids and the combined system GO@surfactant in EOR is evaluated using tests on finely divided limestone in unconsolidated porous medium. Dynamic transport and oil displacement tests are used to assess the systems'' applicability as EOR additives.Acknowledgments: CNPq / Fundação Capes / Petrobras.References:[1] SIKIRU S, Rostami A, Soleimani H, et al. Journal of Molecular Liquids. 321,114519 (2021). [2] SOLDANO, C.; MAHMOOD, A.; DUJARDIN, E. Carbon. 48 (8) 2127-2150 (2010).

Growth and characterization of Bismuth tri-iodide – Graphene 2D material.

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The BiI3-Graphene 2D material was obtained by physical vapor deposition in a specialized equipment designed to operate under pressure conditions of 10-7-10-8 mBar, which allowed for strict control of variables such as source and substrate temperature, deposition time, and source-substrate distance. BiI3 was grown onto both graphene-covered TEM grids and single layer graphene SiO2/Si substrates, previously ozone-treated. The growth conditions included a source temperature of 265 ± 1°C, substrate temperature of 40 ± 1°C, depositiontime of 30-120 s, source-substrate distance of 20.0 ± 0.5 mm, and a high-purity Argon atmosphere. The resulting 2D material layers were characterized using High ResolutionTransmission Electron Microscopy (HR-TEM) and Energy Dispersive Spectroscopy (EDS) forgrids, and X-Ray Reflectometry (XRR) and Gracing Incidence X-Ray Diffraction (GIXRD) forsubstrates.The layers of BiI3 exhibited a rotation between them, resulting in a Moiré effectindicating the presence of BiI3-Van der Waals superstructures within two or three BiI3 layers. The majority of the surface was covered, although not uniformly. Fast Fourier Transforms (FFTs) indicated that the orientation of the layers was always with the c-axisperpendicular to the grid substrate, in its rhombohedral phase R-3. GIXRD confirmed thelayer composition as BiI3 growth onto single layer graphene SiO2/Si substrates, in agreement with EDS and HR-TEM results, and the orientation of the BiI3 layers with the c-axis perpendicular to the substrate, which also agrees with HR-TEM results. The BiI3 layerexhibited a density of around 6.0 g/cm3, a thickness of 34.6 nm, and a roughness of 6.0nm.In the future, it is proposed to perform solar simulation tests to evaluate the potentialphotovoltaic applications of the material.

Investigating Raman scattering of MoS2 monolayer onAu Plasmonic Grating.

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Two-dimensional transition metal dichalcogenides (2D-TMDCs) have attracted attention because of their unique properties compared to their bulk counterparts [1], which make them ideal candidates for optoelectronic devices [2]. MoS2 monolayer (ML) is an example of 2D-TMDCs compound, considered one of this family''s most stable layered materials [1]. It is an inorganic semiconductor with a direct gap and high photoluminescence (PL) emission, where the optical response is primarily determined by excitonic transitions [1]. In addition, MoS2 ML exhibits only three zone-center first-order Raman active modes [3]. It is also possible to observe second-order bands, depending on the energy of excitation [4]. In this study, we investigated the vibrational properties of MoS2 ML deposited on different fabricated Au gratings (GR) - square and circular geometries. These GR are composed of long sub-wavelength slits with a width of 100 nm, separated by varying distances in the nanometer range. We examined the MoS2 ML on both supported and suspended regions inside the GR using confocal μ-Raman spectroscopy. Our results showed that the out-of-plane mode was more intense in the suspended regions than in the supported ones for all geometries. Additionally, we verified that a broad band around 450 cm-1 associated with second-order effects was more pronounced in the suspended regions. Moreover, we observed the Raman modes enhancement due to the plasmonic field in the suspended region and depended on the GR parameters. In conclusion, our results demonstrate a dependent relationship between the vibrational lattice properties and the characteristics of the Au plasmonic GR.

Acknowledgments: FAPESP 2021/03311-3, SNSF 200021L_205114, and SISFOTON-USP 440237/2021-1.

References:

[1] Kolobov, V.A, et al., J., Springer Ser. Mater. Sci. 239., (2016).
[2] Lembke, D., et al., ACSNano 6., (2012).
[3] Pimenta, M.A., et al., Acc. Chem. Res. 48.1, (2015).
[4] Li, H. et al., Adv. Funct. Mater. 22.7, (2012).

Investigating the Electronic Properties of Strained Biphenylene-Based Structures through the DFTB Method

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In 1941 a novel organic compound known as the biphenylene molecule, a hydrocarbon compound consisting of two six-membered aromatic rings separated by a four-membered ring, was proposed by W. C. Lothrop[1]. When connected to form a network, carbon octagons are formed between two biphenylene molecules. Due to recent developments in materials research, the biphenylene network has become a promising allotrope of sp^2-hybridized carbon atoms, distinct from graphene sheets, as it exhibits different electronic properties[2]. The synthesis of a biphenylene network (BPN), was recently achieved[2] through an on-surface interpolymer dehydrofluorination (HF-zipping) reaction. This allotrope presents many possible applications in the field of materials. Our study focuses on examining the effects of strain on its electronic properties, including density of states (DOS), band structure, and energy gap. Our main goal was to evaluate the BPN' s electronictunability by applying strain and whether it remains metallic thereafter. We have also obtained mechanical properties such as its Young' s modulus and concluded that it has a brittle nature, given that it loses its integrity after a certain amount of strain is applied. Toachieve this, we used the DFTB+[3] software to implement the DFTB method for our simulations and discovered that the results yield a metallic behavior for the BPN. As strainis applied, the system begins to show a Dirac point in the Fermi energy, while still maintaining its metallic properties.
Acknowledgments:
We would like to thankthe department of Physics at the Federal University of Parana for the computational and financial support as well as the National Council for Scientific and TechnologicalDevelopment (CNPq) for the financial support.
References:
[1] W. C. Lothrop, J.Am. Chem. Soc. vol. 63 (5), 1187-1191 (1941)
[2] Q. Fan, L. Yan, M. Tripp et al,Science vol. 372, 852-856 (2021)
[3] B. Hourahine et . al., Journal of Chemica

Investigating the Mechanical Behavior of Molecular Kirigamis of WSe2 using Molecular Dynamics Simulations

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Transition metal dichalcogenidesis study, we investigate the mechanical behavior of molecular kirigamis of WSe2 using molecular dynamics simulations with the LAMMPS software [1] and the reax force field [2]. Kirigami is a process of creating new patterns and shapes by cutting, stretching, or twisting materials. In molecular systems, kirigamis can be generated by manipulating the atomic structure of a material to create new molecular arrangements. Our study focuses on investigating the behavior of molecular kirigamis of WSe2 and their impact on the material''s fracture patterns and elastic properties. To minimize modeling stresses, we equilibrate the system with the NPT ensemble for 100 ps and then apply uniaxial tension with the NVT ensemble at 300 K. We simulate the temporal evolution of atoms and molecules in the system using the Newton equations of motion and the Velocity-Verlet algorithm. Similar studies have been conducted on kirigamis of graphene and MoS2 [3], and this work builds on these previous findings to investigate WSe2. Our findings contribute to the fundamental understanding of WSe2, open up new avenues for material science research and emphas the importance of utilizing advanced computational methods to investigate the properties of materials.

Acknowledgements: Thank you to the National Council for Scientific and Technological Development (CNPq), the Foundation for the Support of Research in the Federal District (FAP-DF), the Coordination for the Improvement of Higher Education Personnel (CAPES), and the University of Brasília (UnB) for their support.

References:

[1] PLIMPTON, Steve. Fast parallel algorithms for short-range molecular dynamics. Journal of computational physics, v. 117, n. 1, p. 1-19, 1995.

[2] VAN DUIN, Adri CT et al. ReaxFF: a reactive force field for hydrocarbons. The Journal of Physical Chemistry A, v. 105, n. 41, p. 9396-9409, 2001. [3] HANAKATA, Paul Z. et al. Highly stretchable MoS 2 kirigami. Nanoscale, v. 8, n. 1, p. 458-463, 2016.

Layered double hydroxides for immobilization ofhazardous wastes

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Cementation is a widespread and consolidate technology used to immobilize diverse types of hazardous materials in a stable cement-matrix avoiding its release into the environment. Recently, many low molecular weight organic admixtures are being employed to improve the physical properties of this matrix. During the cementation process toxic oxyanions are intercalated and immobilized in AFm-phases. AFm-phases refer to the family of two-dimensional layered double hydroxides (LDH) and are formed during hydration and curing of cement. The goal of this study is the pure synthesis and precise characterization of solid solution formation between small organic molecules (from non-corrosive accelerators of cement) and inorganic toxic oxyanions like CrO_4^{-2}. Stoichiometric amounts of CaO, $CaAl_2O_4$, $CaCrO_4$and decarbonated water were mixed at a W/C =10 with $Ca(HCOO)_2$at 25°C to investigate the formation of different LDH at different relative humidities. The experiments were conducted in a glove box under the Ar atmosphere to avoid carbonation. X-ray powder diffraction, SEM-EDS, thermal analysis, Raman, IR-spectroscopy and Ion chromatography(IC) were used in order to characterize the hydrated compounds. The X-ray diffraction patterns of $CrO_4^{-2}/(HCOO)_2^{-2}$-containing mixtures show well crystallized AFm-phases with trigonal/hexagonal or monoclinic structures varying with increasing HCOO-concentration. A Pawley fit was performed to determine the lattice parameters of the synthesized compounds and using a combination of EDS/IC the composition of the samples was determined. The spectroscopic measurements confirm the intercalation of $CrO_4^{-2}/(HCOO)_2^{-2}$into the interlayer of the synthesized LDHs. Scanning electron microscopic analysis shows that the morphology of the synthesized samples varies as a function of $HCOO^-$concentration.

Low-Temperature Treatment of Graphene Oxide Dispersions: Implications on its Optical and Electrical Properties

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The excellent physicochemical properties exhibited by carbon nanomaterials, such as high electrical conductivity, good optical transmittance, and exceptional flexibility, make them a major topic of research due to their wide-ranging possibilities for applications in various areas. Among them, graphene oxide (GO) is a two-dimension carbon nanomaterial with hybridization sp2/sp3 and the nanosheets have different oxygenated functional groups randomly distributed, for example, carbonyl (C=O), hydroxyl (C-OH), carboxylic acids (HO-C=O) and epoxy (C-O-C). The presence of oxygenated groups indicates that this material is oxidized and it becomes possible dispersion of the material in water. In this work, the synthesis of different types of graphene oxide was made and aqueous dispersions of these materials were kept under thermal treatment at 80°C for seven days to study the effect ofthe diffusion of oxygenated groups through the plane of the material. This phenomenon previously reported in a paper [1] and little experimentally explored until now. All dispersions were characterized using ultraviolet-visible absorption spectroscopy (UV-VIS). The optical bandgap was determined from the UV-Vis spectra and the results showed that with the passing of the days, a reduction in the bandgap value occurred for all samples. Furthermore, thin films were fabricated using these dispersions, the Hall effect technique analyzed the electrical properties and showed a reduction in the value of sheet resistance for GOs that were subject to temperature. The results indicated that the phenomenon of diffusion is dependent of GO stoichiometric and degree of exfoliation and for this some oxygenated groups are more likely to diffuse in the GO structure and this is in good agreement with the theoretical predictions.

Acknowledgments: The authors thank CAPES, CNPq and FAPEMIG for funding this work.

References: [1]: KUMAR, P. V. et al. Nat. Chem.6, 151-158 (2014).

Magnetic properties of open shell element dopedsilicene

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The growing interest in low-dimensional materials led to the emergence of important ramifications in scientific knowledge, such as the emergence of nanoscience and nanotechnology. Materials at the nanometric scale present different behaviors from those ofa macroscopic structure. Due to associated quantum effects, these materials may have new properties such as high electronic mobility and thermal conductivity, in addition to greater mechanical strength. On the other hand, silicon is a material whose technological application is well established, and obtaining this material in nanostructured form increases its possibility of integration in current technology. Doping with open layer elements can be used to adjust the band gap of silicene and at the same time introduce new functions, such as making the material ferromagnetic. However, several aspects of silicene doping with transition metals still remain unanswered, such as dopant solubility limits, site preference for adsorption and doping, and formation of magnetic domains. The direction of adsorptionof the chromium atom on the silicon monolayer was also investigated, the silicene bilayerwas also investigated with different types of stacking, the doping was done in order to position the chromium atom between the two sheets. In this work we carry out the investigation of diffusion doping of Chromium atoms on silicene. We verified that some metals introduce new magnetic properties in silicene, in addition to forming metallic alloyswith it, which is promising for the application of these materials in electronic and magneticdevices.

Mapping defects in WS_2 monolayers doped withVanadium by Linear and Non-Linear Optics

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Two-dimensional (2D) materials have proven to be very relevant in basic research and technological applications since the discovery of graphene almost two decades ago. Other families of 2D materials, in particular Transition Metal Dichalcogenides (TMD), have different electronic and vibrational properties than graphene and therefore allow us to formulate new technological applications. For example, it has recently been shown that a two-dimensional semiconductor can be transformed into a ferromagnetic material at room temperature (300 K) with the introduction of outer transition metal atoms as dopants [1]. The objective of this work is to study how the addition of these dopants changes the optical and structural properties of these 2D materials. To this end, we use nonlinear optics techniques such as Four Wave Mixing (FWM) in conjunction with linear optical techniques (Raman Spectroscopy and Photoluminescence) to study samples of Tungsten Disulfide (WS_2) doped with different concentrations of Vanadium. The PL spectrum of the doped samples showed two peaks, excitons $A$and $A*$, the last present in the samples with the introduction of Vanadium. Resonant FWM images with exciton $A*$reveal distinct intensities throughout the sample, possibly showing that the introduction of defects is inhomogeneous and concentrated in lines connecting the center to the vertex of the triangular WS2 flakes. The Raman Spectroscopy results also show changes in the vibration frequencies of E_{2g}modes along these lines indicating an increased presence of Vanadium related defects, showing that the introduction of Vanadium is not homogeneous throughout the sample, hence improvements in the growth of these samples need to be made for future technological applications of these compounds.

Acknowledgements:INCT, CNPq, CAPES, UFMG.

References:

[1] ZHANG, Fu et al. Monolayer vanadium-doped tungsten disulfide: a room-temperature dilute magnetic semiconductor. Advanced Science, v. 7, n. 24, p. 2001174 (2020).

Measuring and mapping the interfacial thermal conductance in Graphene/SiO_2 and Graphene/BN interface with pump-probe microscopy

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Ultrafast optical pump-probe is a well know and powerful tool for studying excited-state dynamics in material' s science. In such an experiment, the pump pulse is responsible for creating an out-of-equilibrium configuration in the sample, e.g., exciting carriers to higher energy levels and elevating the local temperature. By detecting the probe pulse, which is delayed back with respect to the pump pulse, we can relate the changes in its transmitted intensity, i.e., transient absorption (TA), with the carrier''s dynamics in our sample. In graphene, the processes induced by the pump pulse can be divided in three parts: I) excitation of electrons, which occurs in a femtosecond scale; II) cooling of these hot electrons creating hot optical phonons (~ 1-2 ps); and III) anharmonic decay of optical phonons into acoustic phonons, which are responsible for the dissipation of heat between the graphene and the substrate, having a much longer lifetime decay (~ 100 ps).[1]Here, by combining optical pump-probe with a laser scanning system, we performed a spatial and temporal resolved pump-probe microscopy in graphene over silicon dioxide (SiO_2) and boron nitride (BN). By measuring the longer lifetime related with heat dissipation of graphene to the substrate, we were able to determine the interfacial thermal conductance of graphene with these substrates and its spatial variation. Our work opens opportunities for testing the heat dissipation of graphene on different substrates and to engineer more sophisticated heterostructures for heat management on future electronic devices.

Acknowledgments:
CAPES, CNPq, FAPEMIG, INCT-Nanocarbono.

References:

[1] MAK, Kin Fai; LUI, Chun Hung; HEINZ, Tony F. Measurement of the thermal conductance of the graphene/SiO 2 interface. Applied Physics Letters, v. 97, n. 22, p. 221904, 2010.

Mid-infrared surface plasmon-polaritons in metallic-dielectric interfaces

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Surface plasmon-polaritons (SPP), quasi-particles originated from the coupling of photons to collective oscillations of free charge carries, are intrinsically two-dimensional (2D) modes of the interface between a material with metallic character and a dielectric insulator. In the technologically attractive mid-infrared (IR) range, SPPs can access and retrieve information from interface states associated to molecular vibrations, electronic transitions of energy around 50 - 300 meV and room temperature thermodynamical reactions. But, in analogy to free space waves or propagating optical modes in fibers, the use of SPPs as information carriers is critically dependent on their propagative properties in media supporting mid-IR plasmonic waves. Despite semiconducting and metallic micro- and nanostructures can bear stationary and localized SPPs enabling functionalities as antennas and resonators in the mid-IR, these media are reported to have high ohmic losses that hamper the efficiency of propagative modes. As a promising alternative, we present here the first real-space, nanoscale resolved imaging/spectroscopy of micrometer-d wavelength SPP waves in insulator/metal/insulator (IMI) heterostructures with propagation lengths exceeding 20 μmat room temperature in the 750-1450 cm^(-1) mid-IR range. Our theory predicts SPP group velocities reaching 20 % of the light velocity in vacuum and 0.2 - 0.4 ps lifetimes. We further demonstrate that the SPP waves interact with polar crystals phonons in the strong coupling regime. As a general effect of the metal/dielectric interface, the mid-IR SPP waves can be compelling for fast metal-based plasmonics, whilst their ability to strongly couple to phonons can be further explored for enhanced sensing in the mid-IR.

Optical Characterization of Vanadium Doped WS_2 Monolayers with Room-Temperature Ferromagnetic Ordering

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The defect engineering of 2D transition metal dichalcogenides (TMDs) has been widely explored in order to tune the properties of these materials and thus expand their applications. For instance, dilute magnetic semiconductors have been achieved by the introduction of spin-polarized transition metal atoms as substitutional defects in TMDs. A long-range ferromagnetic ordering even above room temperature in vanadium doped WS_2 [1] and WSe_2samples was recently reported, shedding light on the large possibilities for spintronic devices fabrication. Hence, the characterization of the optical and electronic properties of these V doped TMD monolayers as well as the understanding of their mechanism of magnetization become crucial. In this work, we have combined both theory and different experimental linear and nonlinear optical techniques to probe the changes in the electronic structure of WS_2monolayers with increasing vanadium doping concentration. Density functional theory band structure calculations showed a down shifting of the Fermi level inside the valence band for the doped samples and the emergence of an occupied V flat band. Power dependent photoluminescence and four-wave mixing resonant profiles revealed the presence of free holes in the valence band edge and a major role of the V flat band in the optical transitions close to the A exciton energy. Moreover, resonant Raman spectroscopy were performed and displayed a blueshfit in the B exciton energy under increasing V doping, while resonant second-harmonic generation measurements showed no modification in the C exciton energy. Besides that wide optical characterization, our experimental results corroborated by the theoretical calculations indicate an indirect mechanism of magnetization for these V doped WS_2monolayers. Furthermore, our work reinforce the potential of distinct optical tools to probe defects in 2D materials.

[1] Zhang, F. et al. *Advanced Science*, 2020, **7**, 24, 2001174.

Optical switching a photon-avalanche-like mechanismin $NdAl_3(BO_3)_4$ particles excited at 1064 nm by an auxiliary beam at 808 nm

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In recent years, an unconventional excitation of trivalent neodymium ions (Nd^{3+}) at 1064 nm, not resonant with ground-state transitions, has been investigated with the unprecedented demonstration of a photon-avalanche-like (PA-like) mechanism, in which the temperature increase plays a fundamental role. As a proof-of-concept, $NdAl_3(BO_3)_4$ particles were used. A consequence of the PA-like mechanism is the absorption enhancement of excitation photons providing light emission at a broad range covering the visible and near-infrared spectra. In the first study, the temperature increase was due to intrinsic nonradiative relaxations from the Nd^{3+} and the PA-like mechanism ensued at a given excitation power threshold (Pth). Subsequently, an external heating source was used to trigger the PA-like mechanism while keeping the excitation power below Pth at room temperature. Here, we demonstrate the switching on of the PA-like mechanism by an auxiliary beam at 808 nm, which is in resonance with the Nd^{3+} ground-state transition $^4I_{9/2} \rightarrow \{^4F_{5/2}, ^2H_{9/2}\}$. It comprises the first, to the best of our knowledge, demonstration ofan optical switched PA, and the underlying physical mechanism is the additional heating ofthe particles due to the phonon emissions from the Nd^{3+} relaxation pathways when excitingat 808 nm. The present results have potential applications in controlled heating and remote temperature sensing.

Optimization of Graphene Oxide Synthesis: influence of the graphite-oxidant ratio on the BET surface area

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Graphene is a nanomaterial composed of carbon atoms forming a hexagonal structure. It is well known that their crystals are the thinnest one and have great commercial interest due to their electrical and mechanical properties. Among the existing synthesis methods, the most used are through chemical routes[1]. The chosen method for this work was the Hummers method[1]because it has several advantages in terms of process efficiency and reproducibility. In the Hummers methodology, graphene oxide (GO) is obtained through an oxidation reaction of graphite, where several π bonds react to incorporate functional groups such as carboxyl, hydroxyls and carbonyls, depending on the intensity of the oxidation reaction, making GO rich in sp^3carbon[2]. The aim of this study was to compare graphene oxides produced using different graphite-oxidant ratios and correlate the material oxidation with its BET surface area. Through characterization by some techniques, it was noticed that the larger the graphite-oxidant ratio, more intense the change in its structure, that is, as higher the proportion between them, more successful the oxidation process[3]. This directly implied on the increase on the BET surface area of the material: increasing the graphite-oxidant proportion from 1:3 to 1:6, one order of magnitude increase in the BET area was observed. Following this logic, it was possible to modify pre-existing methods in the literature in order to find an ideal proportion to reach larger surface areas.

Acknowledgements:
We thank the CNPq for the financial support.

References:
[1] HUMMERS, W. S.; OFFEMAN, R. E. Preparation of Graphitic Oxide. J. Am. Chem. Soc. v. 80, 1339-1339, 1958.
[2] WANG, Hui; HU, H.Y.; Effect of Oxygen Content on Structures of Graphite Oxides. Industrial Engineering Chemistry Research v. 50 (10), p. 6132-6137, 2011.
[3] COTE, L. J; KIM, F.; HUANG, J. X. Langmuir-Blodgett assembly of graphite oxide single layers. J. Am. Chem. Soc. v. 131, p. 1043-1049, 2009.

Optoelectronic properties of MoS_2 devices for hybrid solar cells applications

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Two-dimensional metal dichacogenides have been under intensive research due to the potential applications in optoelectronics devices. Thin film semiconductors, such as Molybdenun disulfide (MoS_2), are a promising alternative semiconductor for hybrid photovoltaic technologies due to its absorption in the near-infrared spectrum, its good charge carrier transport and its ability to easily form 2D structures. In this work we focus on the investigation of optical properties of MoS_2dilluted in ethanol. We have studied the broadband response to ultraviolet, visible and near-infrared (UV-Vis-NIR) range. Our results will be used in the development hybrid solar cells using MoS_2.

Acknowledgments: Fundação Araucária, CNPq and CAPES.

Polycrystalline Aluminum-Doped Silicene: Stability and Mechanical Properties

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Silicene is a promising material due to its potential to have unique electronic, optical and mechanical properties that could be useful in various technological applications. These properties, however, depend on the honeycomb structure of silicene, which has already been shown to have several stability issues. Its monolayer form is only stable when attached to a substrate, which may hinder its application capability. Conversely, its bilayer structure can theoretically be stable in free-standing form, especially when doped. The existing works are still in their early stages and do not consider more realistic structures, such as polycrystalline silicene, which will certainly occur with large-scale synthesis. In this context, this work investigates the stability and mechanical properties of polycrystalline bilayer silicene doped with aluminum. To this end, we applied molecular dynamics simulations using a recently develop ADP potential for Si-Al available in the literature [1]. Polycrystalline bilayer silicenes with AA stacking were created using the Voronoi tessellation with a size of 270 Å x 270 Å containing different percentage of Al, ranging from1 to 4%. The results show that the systems remains stable even at room temperature, withAl atoms deviating from the silicene sheet by 0.7 Å, causing a von Mises shear strain of 0.16in the nearby lattice. However, the doping level does not significantly impact the silicenestrength compared to a non-doped polycrystalline silicene, as the deformation and, consequently, the fracture occurs at the grain boundaries. These results suggest that polycrystalline bilayer Al-doped silicene can be a candidate for electronics applicationswhere some strain levels are required at room temperature.

Acknowledgments:

The authors are thankful for the financial support of the Brazilian agencies FAPERJ and CNPq.

References

[1] S. Starikov et al., Comput. Mater. Sci., 184, 109891 (2020)

Ramam Spectroscopy Studies of $PtSe_2$ and The Formation of Selenium Vacancies Using Temperature

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Platinum diselenide ($PtSe_2$) is a Transition Metal Dichalcogenides (TMD) material with potential for applications on hydrogen evolution, optoelectronic devices, and photocatalytic applications. TMD materials have weak van der Waals interaction between interlayers, which creates a passaway to exfoliate those materials and separate into two-dimensional (2D) nanosheets. Raman spectroscopy is an essential characterization tool for 2D materials with potential to understand defects due to the sensitivity of Raman technique with the breaking of the conservation of momentum rule, i.e. Raman peaks can shift, broaden, change their relative intensity, or additional peaks occur with the presence of defects. The main defects studied for Raman are vacancies, isolated functional groups, substitutional atoms, dislocation, and grain boundaries. In this work, a thin film of PtSe2 was obtained through the selenization of a metallic platinum substrate and Ramam spectroscopy was used to study its structure and stability over a range of temperatures (RT-500°C). Transmission Electron Microscopy (TEM), X-ray Diffraction (XRD), Scanning Transmission Electron Microscopy (STEM), and X -ray Photoelectron Spectroscopy (XPS) characterizations were performed before and after the in-situ Raman temperature studies. It was possible to observe the effects of temperature and the generation of defects of selenium vacancies on the material with the Raman peaks shift to lower energy possibly due to the local atomic change in the structure and the appear of LA band (Longitudinal Acoustic Mode), considered a defect band in Raman Spectroscopy.

Acknowledgments:
We would like to thank the FAPESP agency and the National Center for Research in Energy and Materials (CNPEM).

References:
[1] Gulo, D.P. et al. Sci. Rep. 10, 19003 (2020)

Reduction of MoO3 with organic acids activated by microwave radiation

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Reduced molybdenum oxide (MoO_{3-x}) is an important 2D material with a wide range of applications as catalysis, analytical chemistry and sensors. Its obtention have been made by several methods like solvothermal, hydrothermal and microwave exfoliation [1,2]. Furthermore, it is known that water dispersed nanosheets of MoO3 can be easily reduced by ascorbic acid [1]. Although, bulk molybdenum oxide does not react in that same way. The aim of this work was demonstrated that microwave potentialize the MoO3 reduction by ascorbic acid and activate it reduction by organic acids which presents -COHCOOH structure. For this purpose, bulk and exfoliated MoO3 was placed to react in a water solution of ascorbic acid through 30 min under microwave radiation at 120 °C. Moreover, reaction with organic acids (acetic, succinic, citric, lactic, malic or tartaric acid) were conducted, 1:1 and 2:1 molar ratio of organic acid to molybdenum oxide were evaluated. UV-Vis measurements presents absorption bands centered at 690, 800 and 1000 nm related to Mo6+ reduction to Mo5+ and Mo4+ [3] when reacts with ascorbic acid, lactic acid, malic acid or tartaric acid. Further, the samples were characterized by SEM, FTIR, Raman and XRD. A new mechanism based on molecule polarization by microwave radiation was proposed to explain those results. Steric effect supports the unreactive of MoO3 with citricacid.

Acknowledgments

This work was supported by: CNPq, CAPES and FACEPE.

References

[1] Li, R., An, H., Huang, W., He, Y. (2018). Sens. Actuators B Chem., 259, 59-63.
[2] Wei, Z., Gasparyan, M., Liu, L., Verpoort, F., Hu, J., Jin, Z., Zhuiykov, S. (2023). J. Chem. Eng., 454, 140076.
[3] He, T., Yao, J. (2003). J. Photochem. Photobiol.: Photochemistry Reviews, 4(2), 125-143.

Second-Harmonic Generation enhancement of monolayer molybdenum disulfide from gold plasmonic gratings

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Monolayer transition-metal dichalcogenides (TMDs), with unique electronic and optical attributes not found in their bulk forms, have recently attracted significant attention from the scientific community [1]. In particular, due to their non-centrosymmetric nature and relatively large second-order nonlinear susceptibility, these materials can exhibit an unusual nonlinear optical response [2]. However, the atomic thickness of these monolayers limits the interaction between the TMDs and light, generally resulting in an inefficient second-harmonic generation (SHG) process [3]. Here, we demonstrate SHG enhancement of MoS_2monolayer via surface plasmons generated at square gold gratings composed of 20 μm long sub-wavelength slits with a width of 100 nm and separated by distances varying from 1000 nm to 300 nm. The SHG signal from the hybrid device is three times greater than the signal from MoS_2deposited on flat gold and does not show peak broadening in the spectra. Additionally, the dependence of SHG intensity on excitation power showed two distinct regimes. A slope of ~ 1.9 was observed for low excitation powers, revealing a quasi-quadratic dependence consistent with the second-order nonlinear process. For high excitation powers, the slope is reduced to less than one, indicating saturation of the SHG process. Our work sheds light on the physical mechanisms of SHG in MoS_2-plasmonic nanostructures and may help the development of efficient integrated nonlinear devices.

Acknowledgments:
This work was supported by Brazilian CAPES agency grant 88887.609043/2021-00, FAPESP grant 2021/03311-3 and Swiss National Science Foundation grant 200021L_205114.

References:
Wang, Qing Hua, et al. Nature nanotechnology 7.11 (2012): 699-712.
You, J. W., et al. Nanophotonics 8.1 (2018): 63-97.
Lin, Xianqing, et al. ACS nano 12.1 (2018): 689-696.

Strain engineering of MoS2 monolayers onto highly stretchable electrodes for electrochemical hydrogen production

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Molybdenum disulfide has been considered a promising material for application as an electrocatalyst for hydrogen evolution. However, in its natural form, it has poor activity, requiring defect engineering steps through methods such as chemical etching and thermal annealing[1] to activate its basal plane. Associated with the creation of defects it was demonstrated that by applying mechanical strain to MoS2 layers it is possible to further increase its performance due to the modulation of its band energies. Typically, most of the works studying the influence of strain on MoS2 activity employ rigid substrates containing microcone structures of fixed sizes and diameters, limiting the deformation to a certain value. On the other hand, using an elastomeric substrate for the fabrication of modified electrodes makes it possible to tune the strain at different levels in the same device, in addition studies of deformation cycles can be carried out. With these considerations, this work presents initial results related to the development of a polydimethylsiloxane-based stretchable electrode for later application in the development of functional electrodes based on MoS2. After the fabrication of the electrode, bulk MoS2 is transferred onto the electrode's surface and electrochemically thinned in 0.5 M H_2SO_4 solution in a second step[2]. The ultra-large MoS2 monolayers were characterized by Raman and photoluminescence. Chemical etching using H_2O_2 and the formation of the corrugated surface will be shown.

Acknowledgements:
FAPESP Grant number 2022/00955-0, Brazilian Center for Research in Energy and Materials

References:

[1] L. H. Hasimoto, J. Bettini, E. R. Leite, R. S. Lima, J. B. S. Junior, L. Liu, M. Santhiago, ACS Applied Engineering Materials 1, 2, 708-719 (2023)

[2] N. Freitas, B. R. Florindo, V. M. S. Freitas, M. H. de O. Piazzetta, C. A. Ospina, J. Bettini, M. Strauss, E. R. Leite, A. L. Gobbi, R. S. Lima, M. Santhiago, Nanoscale, 14, 6811 (2022)

Study of properties of MoS2 and PEDOT:PSS compositein different concentrations

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The objective of this work is to study the morphological and electric properties of the composite made from the mixture of MoS2 and PEDOT:PSS in different proportions, using water as a solvent. PEDOT:PSS is a polymeric material made from two different polymers, poli(3,4-etilenodioxitiofeno) (PEDOT) and polystyrene sulfide (PSS), due to its high conductivity is commonly used in electrical devices such as organic photovoltaics (OPVs), sensory gases and organic thermoelectric (TE) [1], [2]. MoS2 is a semiconductor that can be exfoliated in a bidimensional material, highly used in the industry as a lubricant due to its weak interaction between its layers (Vander Walls force). Due to its electric properties, 2D-MoS2 is being used in other applications, for example, used as an electrode in a solar cell [3]. The mixtures were made using the 2D-MoS2 as a base and then doing the percentage of PEDOT:PSS, varying the sample from 10% to 45%. To do the morphology analyses we obtain images from Scanning Electron Microscope (SEM), Transmission Electron Microscopy (TEM) and Atomic Force Microscopy (AFM). The electric characterization was made from the 4 probe method, which showed us a significant improvement in the electric properties up to 2 orders of magnitude if compared to the pure PEDOT:PSS. Acknowledgments: This work was supported by CNPq, CAPES, PIPE - UFPR,UFMG.

References:

[1] Z. Fan and J. Ouyang, “Thermoelectric properties of pedot: Pss,” Advanced Electronic Materials, vol. 5, no. 11, p. 1 800 769, 2019.

[2] M. F. F. das Neves, J. P. V. Damasceno, S. Holakoei, et al., “Enhancement of conductivity and transmittance of graphene oxide/pedot: Pss electrodes and the evaluation of charge transfer dynamics,” Journal of Applied Physics, vol. 126, no. 21, p. 215 107, 2019.

[3] A. B. Kaul, “Two-dimensional layered materials: Structure, properties, and prospects for device applications,” Journal of Materials Research, vol. 29, no. 3, pp. 348-361, 2014.

Study of the synthesis of reduced graphene oxide obtained from organic sources and its application in polymeric composites

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Graphene, an exceptional nanomaterial, has garnered significant attention due to its extraordinary properties and potential applications. This ongoing study aims to conduct a comparative analysis of reduced graphene oxide (rGO) synthesized via the modified Hummers'' method from graphite and via thermochemical reduction from sawdust, for incorporation into a polyvinyl chloride (PVC) matrix. The synthesized rGO samples will undergo characterization using Raman spectroscopy, with the aim of identifying G and D peaks that have values of width at mid-height greater than 30cm^{-1}. This will confirm the presence of functionalized graphene or graphene oxide. In addition, scanning electron microscopy (SEM) will be utilized to analyze the lateral thickness of the flakes, which contributes to determining the amount of stacked graphene layers present in the synthesized sample. The thickness of ~0.34nm corresponds to a single graphene layer. A comparison of the rGO samples will be conducted to demonstrate the feasibility of using sawdust as a sustainable alternative precursor for graphene synthesis, with the aim of reducing the amount of this waste product. In Brazil, it is estimated that 30 million tons of sawdust are generated each year [1]. Subsequently, the synthesized rGO will be incorporated into a PVC matrix [2]. The resulting nanocomposites will undergo characterization for their mechanical and thermal properties, using tensile testing and TGA, to confirm the possible application of rGO synthesized from wood sawdust.

[1] SILVA, Lisiane. Síntese e caracterização de grafeno produzido a partir da serragem de madeira. Alegrete: Unipampa, 2020.
[2] MINDIVAN F.; GÖKTAŞ M. Preparation of new PVC composite using green reduced graphene oxide and its efects in thermal and mechanical properties. 2020.

Study of two-dimensional molybdenum composite and graphene in gas sensor device

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Gas sensing devices based on two-dimensional (2D) materials have excellent gas detection performance. The two-dimensional structure of 2D MoO_3:graphene nanocomposites has been studied for this purpose, as it has a large specific surface area, which increases the adsorption capacity of the gas [1]. The graphene layer acts as a conductive platform that facilitates the transfer of electrons from the $MoO_3$to the gas molecules, leading to a change in the electrical properties of the device [2]. The high sensitivity, selectivity and stability of 2D-MoO_3:Graphene gas sensing devices make them promising candidates for practical gas detection applications. This work describes the characterization of the two-dimensional composite and its application in the active layer of an alcohol gas sensor device. The tests were carried out in ambient atmosphere, under variation of alcohol gas concentration (ppm) and temperature (20 - 120 ºC), with the objective of verifying the response to resistance variation, that is, gas detection in different scenarios.

Acknowledgment:
This work has been supported by CAPES and CNPq.

References:

[1] XH, Liu; TT, Ma; N, Pinna; J, Zhang.Two-Dimensional Nanostructured Materials for Gas Sensing. Advanced Functional Materials, vol. 27, page 37, 2017.
[2] Tabata H, Sato Y, Oi K, Kubo O, Katayama M. Bias- and Gate-Tunable Gas Sensor Response Originating from Modulation in the Schottky Barrier Height of a Graphene/MoS2 van der Waals Heterojunction. ACS Appl Mater Interfaces, vol. 10, page 44, 2018.

Synthesis and characterization of MIL-100(Fe)@GO and MIL-100 (Fe)@rGO nanocomposites

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Metal-organic frameworks (MOFs) are porous structures built from the coordinative bond between metal ions and organic ligands. One of the unique characteristics of MOFs is their permanent porosity. Modifications to its geometry, length, aspect ratio, and functional groups can adjust the , shape, and inner surface properties of a MOF for a variety of applications. However, certain interferents, such as limited thermal and mechanical stability, led scholars to associate MOFs with carbon-based materials, such as graphene oxide and reduced graphene oxide [1]. Thus, this study aimed at the synthesis and characterization of MOF@GO and MOF@rGO nanocomposites. Therefore, the graphene derivatives, GO through the modified Hummers method, and the rGO from the chemical reduction using Ascorbic Acid (L-AA) were obtained. The MIL-100(Fe)@GO and MIL-100(Fe)@rGO nanocomposites were obtained using the solvothermal method. The synthed materials were characterized by X-Ray Diffraction (XRD), Infrared Absorption Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA) and Raman spectroscopy. The Raman spectra confirmed the obtaining of GO and rGO, which had their average number of layers estimated from the broadening of the diffraction peaks referring to the basal plane and the Scherrer equation. XRD data also confirmed the crystallization of MOFs in the composites. The SEM images of the nanocomposites suggest the formation of MOF crystals randomly anchored and dispersed on the surface of GO and rGO. Thermogravimetry data indicate that both nanocomposites have three mass loss events, however the MIL-100(Fe)@rGO system was more stable.

Acknowledgements:UFPE, FACEPE for promoting the scholarship and the SUPRAMMat research group.

References:

[1] FENG, X. et al. Metal-organic framework MIL-100(Fe) as a novel moisture buffer material for energy-efficient indoor humidity control. Building and Environment, v. 145, p. 234-242 (2018)

SYNTHESIS, CHARACTERIZATION AND APPLICATIONOF SP2 CARBON FILMS BY THE CVD METHOD WITHOUT CATALYTIC SUBSTRATES

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This work aims to study graphene synthesis via Chemical Vapor Deposition - CVD, without the use of metallic catalysts. We were able to grow the films directly on a SiO2/Si substrate (300 nm oxide thickness). We demonstrate that the growth of graphene without catalysts is not self-limiting, that is, the film thickness is strongly dependent on the growth time. We investigated different deposition times and observed differences in film thickness and covered area. With longer synthesis time, we observed that the films tend to be less crystalline, despite the better coverage. In order to obtain more crystalline films with fully covered substrates, we developed a CVD growth method called "pulsed gas CVD growth". This method is innovative, as we are not aware of other works in the literature with its application. The pulsed gas flow consists of a growth with curing times throughout the deposition, when the insertion of the carbon precursor (CH4) is interrupted for interspersed periods during the growth. The characterization of our films was performed by scanning electron microscopy (SEM), Raman spectroscopy and electrical characterization by Hall Effect. We observed that the material we synthed is p-type doped, which is in agreement with other works in the literature [1]. This fact is probably related to the CO and CO2 impurities present in the sample. To exemplify in practice the interest of this type of synthesis, we built a field effect transistor using samples grown with continuous and pulsed gas flows. We observed the variation of the Fermi level, the increase of the current and the conductivity of the synthed materials.

Acknowledgements:
The authors of this work would like to acknowledge: Capes, CNPq, Fapemig and the UFJF Nanosciences and Nanotechnology Group (Nano).

References:

[1] A.N. Barbosa et al. Diamond and related materials, v. 95, p. 71-76, 2019.

The influence of graphene addition on the properties ofa styrene acrylic decorative paint

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Coatings are regularly used for decorative purposes and for surface protection. The coatings industry, like many other, is constantly researching and developing new technologies for the formulation and application of new and better coatings. Since 2004, when the graphene (GO) nanomaterial was first isolated, scientists in the coatings industry have been looking for ways to use GO as an additive to improve the performance and technology of coatings in different application areas. GO enables a wide array of functional coatings, for many possible applications. GO is a single, tightly packed layer of carbon atoms arranged in a hexagonal honeycomb lattice. It is the thinnest compound known to man at one atom thick, the lightest material known, the strongest compound discovered. Incorporating GO into a decorative paint can increase its chemical and mechanical resistance and offer greater surface protection. Therefore, different percentages of GO (1.0; 1.5 and 2.5 %) were added in the formulation of a styrene acrylic decorative paint with an eggshell finish. The addition of GO was performed by milling the concentrate with zirconium spheres for 40 min followed by adding the other raw materials by mechanical dispersion using a Cowles-type disc for 20 min. The properties of the standard paint and with different percentages of GO were measured through Konig hardness (ABNT NBR 14946), washability (ABNT NBR 15494), contact angle and surface energy (ramé-Hart 250 goniometer), rheology (Haake ViscotesteriQ, Thermo Fischer Scientific Inc.), and staining tests (coffee, ketchup, oil and wine). The obtained results were promising, with gains in properties, increasing the hardness and washability resistance of the coating, hydrophobic characteristics, as well as improving resistance to staining.

Acknowledgements: The authors would like to acknowledge Anjo Tintas for the financial support, for supplying the paint base and for making the R D laboratory available to carry out the tests.

Theoretical investigation of Graphenylene Nanoflakes stacking effects on optical and electronic properties

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In the last decades, the study of two-dimensional materials has sparked great interest in the scientific community. Graphene, synthed in 2004, has paved the way for advancements in nanoscience and nanotechnology. One of the promising materials in this field is graphenylene, a two-dimensional porous carbon-based material with a structure analogous to graphene and interesting electronic and optical properties. In this context, computational simulations have been instrumental in investigating the properties of these materials at the nanoscale. In this work, the DFTB approximation was used to simulate the stacking of passivated graphenylene nanoflakes. We analyzed different s and shapes of flakes, investigating their optical and electronic properties. Our simulations indicate that stacking of graphenylene nanoflakes can modify optical and electronic properties such as absorption, electronic gaps and the frontier orbitals. Notably, the intensity of absorption peaks decreases as flakes of the same type are stacked. This behavior suggests an interaction between the flakes in the stacking, thereby influencing light absorption and electronic behavior in comparison to monolayer nanoflake. This study can contribute to the advancement of knowledge about the behavior and properties of stacked graphenylene nanoflakes. The performed computational simulations provide valuable insights into the relationship between the stacking structure and the optical and electronic characteristics of these systems. Therefore, the obtained results can be useful in the development of future applications.

Acknowledgements:
G. Batista acknowledges CAPES for the financial support - finance code 001, and R. Paupitz acknowledges the funding agencies: FAPESP (grant #2018/03961-5 and 2021/14977-2), CNPq (grants #437034/2018-6 and #315008/2020-2). Additionally, this work utilized the computing resources of NCC/GridUNESP and CENAPAD-SP.

TMD STRUCTURES GROWN BY CVD ON SP2 CARBON SUBSTRATES

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Low-dimensional carbon-based materials have been the subject of several studies in the last two decades due to their physical properties[1]. In particular, graphene has been widely used in various fields, and it can be synthed by many methods, such as chemical vapor deposition (CVD), which is an efficient method to obtain large-scale and high-quality graphene. A lot of studies have been made regarding CVD growth using metal foils as catalysts. However, for device applications, we have to transfer the as-grown graphene onto another suitable substrate from the metal catalyst surface which needs a series of processes that sometimes may compromise sample quality. Recently, some work has been done towards the direct growth of graphene by CVD on desired target substrates [2,3]. The posterior growth of other materials on top of graphene, such as transition metal dichalcogenides (TMDs), can provide heterostructures that could be interesting in nanoelectronics. In this work, we propose to obtain TMDs or HS grown directly on a sp2 carbon film, all structures grown by CVD method on top of a Si/SiO2 substrate, avoiding any transfer process. We perform spectroscopic measurements to compare these samples with transfer-based carbon-TMD HS in order to infer the differences in layer interaction for both structures.

References:
[1] SANTOS, R.M. Estudo das propriedades eletrônicas de nanomateriais 2D na presença de defeitos estruturais, (2021).
[2] A.N., BARBOSA, F. PTAK, C.D. MENDOZA, M.E.H. MAIA DA COSTA, F.L. FREIRE, Direct synthesis of bilayer graphene on silicon dioxide substrates, Diam. Relat. Mater. 95 (2019) 71-76.
[3] H. Bi, S. Sun, F. Huang, X. Xie, M. Jiang, Direct growth of few-layer graphene films on SiO 2 substrates and their photovoltaic applications, J. Mater. Chem. 22 (2012) 411-416, https://doi.org/10.1039/c1jm14778a.

Trimetallic layered double hydroxides with the composition $[Zn^{2+}_{6-x}M^{2+}_{x}Al_{3}(OH)_{18}][(SO_{4})_{2}Na]\cdot yH_{2}O$ (M^{2+}= Co, Mn).

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Synthetic layered double hydroxides (LDHs) with the ideal composition [M2+6Al3(OH)18][(SO4)2Na]·12H2O were recently described [1,2]. To expand this class of compounds, trimetallic LDHs with composition [Zn6-xM2+xAl3(OH)18][(SO4)2Na].yH2O (M2+=Co, Mn) (x=0 to 6) were synthesized. Solutions of the metal sulfates of M2+, M3+ and Na2SO4 (molar ratios of 6:3:1), with different proportions between Co2+ and Zn2+, were titrated with a 1 mol L-1 NaOH solution until a pH = 9-9.5. The materials in the presence of the mother solution were treated at 90 °C for 120 h, centrifuged at 4500 rpm, washed several times with water after redispersion, and dried at 50 °C for 72 h. X ray diffraction indicated crystalline phases with basal spacing close to 11 Å, attributed to the intercalation of SO42- in a double layer arrangement in the presence of hydrated Na+. A reduction in the basal distance was observed when Zn is replaced by Mn or Co, according to the reduction of the ionic radius. The ICP-OES analyzes confirmed the expected compositions and FTIR analyses showed bands attributed to the O-H and M-O bond, S-O bands of sulfate, in addition to a linear shifting of the M-O band due to the replacement of Zn by Co or Mn, according with Vegard's law [3], which states that a linear correlation should be observed when metals with different ionic radii are used in a solid solution. This new trimetallic LDHs, which can exchange cations, anions and even both simultaneously, present potential applications in different areas of science and technology.

Acknowledgements:This study was financed by CAPES - Finance Code 001, CNPq (FW: 300988/2019-2) and FINEP. ARS thanks CNPq for the Postdoctoral fellowship (163817/2020-0) and VVSM thanks CAPES for the Ph.D. scholarship.

References:[1] Sotiles, A.R.; Baika, L.M.; Grassi, M.T.; Wypych, F., J. Am. Chem. Soc. 141, 531 (2019)[2] Gomez, N.A.G.; Sotiles, A.R., Wypych, F., Appl. Clay Sci. 193, 105658 (2020)[3] Vegard, L., Zeitsch. Phys. 5, 17 (1921

Two-dimensional oxide structures of perovskitestructure via sacrificial layer dissolution

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Recently, Two-dimensional oxides and their application in flexible and wearable electronics films have been extensively studied, due to their potential applications in sensors, biomedical and electronic devices. So, there is an effort to development of different synthesis routes to obtain freestanding inorganic films, which can be manipulated and used to build new functional electronic devices, aiming to reach a synergy between the greater degree of mechanical freedom and the amplification of the performance of well-known functional oxides. In this work, we report the fabrication of freestanding RNiO3 (R= La and Nd) thin films using NaCl and Sr3Al2O6 (SAO) as sacrificial layers. The NaCl layer were grown using thermal deposition method and the SAO were deposited through a chemical solution deposition (CSD) method, both films were grown on SiO2/Si(100) substrates. Over these sacrificial layers were deposited 1 to 3 layers of an R-Ni precursor solution and each layer were heat-treated at 700 °C in oxygen atmosphere to obtain RNiO3 heterostructures. The structural and microstructural characteristics of these films were evaluated by X-ray diffraction, Raman spectroscopy and scanning electron microscopy. The X-ray results revealed the formation of the NaCl phase, whereas the SAO phase was only reached at a temperature of 1000°C. An initial analysis suggested that de NdNiO3/SAO/SiO2/Si(100) film crystallized in the desired phase. Although, preliminary solubility tests showed that de SAO films were not water-soluble, they seem to be slightly soluble in basic solution. The NaCl films present high solubility in water. The NdNiO3/NaCl/SiO2/Si(100) films present a rough surface, but it seems to present a homogeneous and crack-free film. The electrical resistance as a function of temperature is going to be evaluated of the film over the sacrificial layers and freestanding.

Water Structure and Dynamics inside Two-Dimensional MoS2

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Computational studies on confined water have advanced the field of materials science and nanofluidics in the last decades. They provide a detailed understanding of the water properties and how it is influenced by the geometry and nature of the confining materials. Particularly, MoS2 nanochannels have been proposed as a prominent water sieve, with high selectivity and water flow. Two-dimensional MoS2, with a combination of hydrophobic and hydrophilic sites were shown to even exceed the water flow rate found in pristine graphene. This highlights the importance of studying these materials. Here, we propose a Molecular Dynamics (MD) study of water confined between monolayers composed of MoS2 and graphene oxide using the LAMMPS software. The results show a water organization in layers depending on the distance between the solid-state plates. The structuration also depends on the material and the level of oxidation of the graphene. The results highlight how nanoconfined water behaves differently from bulk and the possibilities for its application.

Y-Advanced Materials - Poster Session

Magnetic properties at high H/T regimes

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Magnetic materials under high applied magnetic fields and at low temperatures present unusual electronics, thermal and magnetic properties that are governed by the H/T ratio. The generation of magnetic fields above the limits imposed by commercial superconducting solenoids is not a simple task, demanding for complex infrastructures and substantial financial support. Fortunately, there are few laboratories, like the National High Magnetic Field at Tallahassee -NHMFL, that are open for the magnetism communities worldwide allowing the access to large H/T regimes. In this presentation, I will review some former experimental results [1-4] I was involved with as well as to discuss some experiments that are underway.

Acknowledgments: Many thanks to my collaborators along the years and to the NHMFL for providing full support to our research work. Financial support from FINEP, CNPq, CAPES and FACEPE are also acknowledged.

References:
1 - A. Franco, F. L. A. Machado, V. S. Zapf, Magnetic properties of nanoparticles of cobalt ferrite at high magnetic field, J. Appl. Phys. **110**, 053913 (2011).

2 - F. L. A. Machado, J. M. Soares, O. L. A. Conceição, E. S. Choi, L. Balicas, Magnetic properties of the nanocomposite $CoFe_2O_4$/FeCo-FeO at a high H/T regime, J. Magn. Magn. Mater. **424**, 323 (2017).

3 - D. R. Ratkovski, L. Balicas, A. Bangura, F. L. A. Machado, S. M. Rezende, Thermal transport in yttrium iron garnet at very high magnetic fields, Phys. Rev. B **101**, 174442 (2020).

4 - M. Cabrera-Baez, K. V. R. A. Silva, P. R. T. Ribeiro , D. R. Ratkovski , E. L. T. França , A. Flessa-Savvidou, B. Casas, T. Siegrist, L. Balicas , S. M. Rezende, F. L. A. Machado, Giant magnetoresistance in the crystalline YCd_6intermetallic compound, Phys. Rev. B **107**, 144414 (2023).

Stabilizing skyrmions on ordered arrays of nanodomes and nanodisks.

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Magnetic skyrmions are non-trivial spin textures that resist external disturbances and are promising candidates for the next generation of magnetic recording devices. However, a major challenge in the realization of devices based on skyrmions is the stabilization of ordered arrangements of these spin textures under ambient temperature and zero applied field conditions. In this work we study the formation and stabilization of magnetic skyrmions in Pt/Co/Ta multilayers. We also demonstrate for the first time the formation and stabilization of magnetic skyrmions on arrays of self-assembled hexagonal nanodomes [1]. Magnetic force microscopy (MFM) images of the arrays of nanodomes with 100 nm shows isolated skyrmions at remanence, as the special topography helps to stabilize the skyrmions in the hexagonal arrangement of the nanodomes. We also investigate the effect of constriction on the nucleation behavior of skyrmions in nanodisks with different diameters, fabricated by electron beam lithography. These systems have been characterized by MFM by MOKE, and micromagnetic simulations were compared with experiments to determine the correlation of the domain textures with the topography of the samples and with the applied field and magnetic parameters of the multilayers.

[1] Tejo, F., Toneto, D., Oyarzún, S., Hermosilla, J., Danna, C. S., Palma, J. L., da Silva, R. B., Dorneles, L. S., Denardin, J. C. (2020). ACS Applied Materials and Interfaces, 12(47). https://doi.org/10.1021/acsami.0c14350

Tailoring ultrathin film growth for integration in high performing magnetic sensors

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Nanoelectronics combines physical principles of materials with the impressing capability of engineering ultra-small devices at the nanoscale. Magnetic field sensors, in particular, magnetoresistive MR sensors, were driven by the technological push from computers and information storage in the early 1990's. Presently they have a mature and transversal levelof implementation in the market, from automotive to biomedical domains. In this talk, I willfirst introduce key concepts in spintronics and highlight the physics mechanisms defining the sensor performance and the figures of merit for the classification of an outstanding MRsensor.
The materials discussed include several varieties of thin films: oxide films as tunneling barriers, ultrathin amorphous and crystalline films, ultrathin textured layers with grain size control, magnetically soft layers or antiferromagnetic films - all combined onto multilayer stacks, typically thinner than 60nm. In addition, the noise mechanisms (the "killing factor" that limits the MR sensor performance) will be discussed, and I will showsuccessful strategies for improving the signal-to-noise ratio, improving the ultimate field detectable by an MR sensor.

The detection principles, sensor design, simulations and experimental validation will be discussed for exciting applications where MR sensors bring added value over other competing technologies. Further, I will show how challenging applications have identified creative solutions, requiring joint skills in transversal areas as physics, materials, electronics and mechanical engineering. I expect academics and engineers to be encouraged to propagate their expertise in magnetism to the young, talented people we see every day, so to trigger innovation in future spintronic sensors.

Acknowledgements: IEEE Magnetics Society Distinguished Lecture program, H2020- FTI-870017-MAG-ID, H2020-MSCA RISE- 2016-grant 734801-Magnamed, H2020-ECSEL-IA-876190-Moore4Medical, FCT- PTDC/NAN-MAT/4093/2021.

Assessing the relaxation mechanisms contributions through the magnetoimpedance effect in YIG/W bilayers

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To study the effect of spin pumping (SP) through the variation of the thickness of the ferromagnetic material (FM) it is important to take into account the two magnon scattering process (TMS). These two effects are known as magnetic relaxation mechanisms and can be estimated through the damping parameter that carries information from intrinsic and extrinsic relaxation mechanisms. One way to evaluate these mechanisms is to analyze, through ferromagnetic resonance (FMR), the line width (ΔH) and consequently the effective damping parameter (α_{eff}). This type of analysis can be done through a traditional FMR experiment, where a fixed frequency is associated with the resonant cavity, or through more sophisticated experiments. For example, using a vector network analyzer (VNA) that allows investigation not only by varying the angle and amplitude of the magnetic field, but also by sweeping the probe frequency. As an alternative to overcome disadvantages in the context of technological applications, we adopted the magnetoimpedance (MI) technique to evaluate ΔH and α_{eff}. However, this choice brought the challenge of performing an electrical measurement on an insulating material, in this case yttrium iron garnet ($Y_3Fe_5O_{12}$- YIG). This material is a ferrimagnetic insulator that has a low damping parameter that makes it a promising candidate for exploring magnetization dynamics. In this work, we evaluate the role of TMS and SP in magnetization relaxation by estimating α_{eff}through the MI effect in YIG/W bilayers, where W is tungsten. By modifying the thickness of the YIG, we explored the contributions of such extrinsic mechanisms to $\alpha_{eff}$and their contributions to the MI response. Our results reveal that the sample geometry allows us to manipulate the magnetoimpedance response over a wide range of frequencies and magnetic fields by mapping the contribution of SP and TMS to the effective damping parameter.

Correlation between interfacial atomic intermixing and the formation of skyrmions

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Among the novel two-dimensional topological magnetic structures investigated in the literature, skyrmions are nanostructures which have large application in the design of new technologies, specially storage devices. One of the main interactions known to be responsible for its stabilization is the Dzyaloshinskii-Moriya interaction (DMI), which arises in the presence of broken inversion symmetry and its strength is related to a large spin-orbit coupling (SOC). Although, asymmetric interfaces which combine magnetic and heavy elements are of great interest in the search for skyrmions, recent experimental works put symmetrical interfaces in perspective as candidates to host topological nanostructures with the observation of room temperature skyrmions in Pd/Co/Pd multilayers. A possible origin for a non-null DMI in these symmetrical stackings is the existence of interfacial interdiffusion, where intermixing of atoms might occur during the deposition process, specially in thin films grown by magnetron sputtering. In this work, using real-space first-principles calculations [6] together with atomistic spin dynamics [7], we investigate the Pd/Co/Pd multilayers, focusing on correlating interfacial atomic intermixing and the formation of skyrmionic phases. Here, through ab initio calculations, we show that the DMI strength is strongly enhanced by the presence of defects, due to a local symmetry breaking induced in the system. Besides that, through atomistic spin dynamics simulations we explore the different magnetic phases and the effects of PMA through a phase diagram and our results indicate that a larger DMI, compared to the pristine case (without defects) can stabilize metastable skyrmions in Pd/Co/Pd multilayers.

References

[1] J. Brandão et al., Sci. Rep. 9, 4144 (2019).
[2] S. Frota-Pessôa, Phys. Rev. B 46, 14570 (1992).
[3] B. Skubic et al., J. Phys. Cond. Matt. 20, 315203 (2008).

Funding Acknowledgement: This work has been supported by FAPESP, CNPq, CAPES and FAPERJ.

Current-induced modifications of superconducting Nb multiterminaltransport bridges with different angles

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In the early days of microelectronics, electromigration was a significant source of failure in circuits. However, it has been shown that, by fine-tuning the threshold current density that triggers this phenomenon, one can modify - in a controllable manner - the material properties at an atomic scale. In this work [1], we investigate the localized material modifications produced by electropulsing on Al capped Nb microbridges with multiterminal configuration. The affected regions on the Nb/Al bilayer terminals are revealed by an in-lens secondary electrons detector in a scanning electron microscope as well as by Kelvin-probe force microscopy, both suggesting a decrease of the work function in the modified areas. In contrast to that, the affected areas are neither apparent through an Everhart-Thornley secondary electrons detector nor through atomic force microscopy, which indicates little morphological changes on the microstructure. These results are elucidated by complementary finite element modelling which permits us to estimate a threshold temperature of (435 ± 35) K needed to induce material modifications. In addition, we demonstrate that the extension of the electroannealed regions is strongly influenced by the terminal geometry, resulting in weak links which affect the superconducting transition. These findings provide further insights on the subtle modifications produced by gentle electroannealing of Nb/Al microstructures and represent a step forward towards mastering this emerging nanofabrication technique.

Acknowledgement
This work was partially supported by FAPESP (grant 2022/03124-1), CNPq (grant 316602/2021-3), and CAPES (Finance Code 001).

References
[1] S. Marinkovic et al. Effect of moderated electropulsing on Nb multiterminal transport bridges. Physical Review Applied, in press (2023).

Effects of Local Spin-Density Waves in Antiferromagnetic phases in Cr-V Alloys

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The magnetic behavior of Cr and its alloys still poses questions related to the itinerant magnetism described by spin-density waves (SDW) [1]. The introduction of small amounts of V in Cr (up to 0.4 at%V) leads to the formation of localized magnetic moments. In the paramagnetic phase, a Curie-Weiss behavior is observed for magnetic fields up to 10 kOe [2,3] giving an evidence of local magnetic moments. The origin of localized magnetic moments in Cr-V alloys can be explain based on the formation of local SDW. A rearrangement of SDW resulting on a short range order, around impurities and different kind of defects, were shown to lead to a self-redistribution of the charge and spin densities in their vicinities [4]. In this present work we reported the observation of magnetic irreversibility associated with local SDW in Cr 0.4 at.%V. The DC magnetic susceptibility measurements were measured using a SQUID magnetometer MPMS-5S from Quantum Design. The temperature dependence of magnetization (M) was measured in the range 5 · T □ 400 K, with increasing temperature, after cooling in zero magnetic field (H) (ZFC), and follow by decreasing temperature, under field cooling (FC), at the same applied magnetic field. The results show irreversibility between the FC and ZFC curves that decreases under applied magnetic field. This behavior is associated to the local SDW due to a magnetic anisotropy directly affecting the long-range order of SDW.

Fabrication and measurement of superconductingqubits

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In recent years, the development of techniques to observe non-classical behavior in nano-scale mechanical structures has brought significant attention due to their potential for fundamental studies of quantum mechanics and new applications such as sensing weak forces and quantum communication [1]. One important approach is through the coupling of nanomechanical resonators with superconducting qubits, which recent experiments have started to develop as tools to manipulate and measure quantum states of mechanics [2]. We are interested in transmon-type superconducting qubits for read-out and control of nanomechanical resonators. This talk will cover preliminary system designs and our efforts to develop transmon qubits coupled to microwave cavities.

[1] Braginsky, V. B. Khalili, F. Y. Quantum nondemolition measurements: the route from toys to tools. Rev. Mod. Phys. 68, 1-11 (1996)

[2] Arrangoiz-Arriola, P., Wollack, E.A., Wang, Z. et al. Resolving the energy levels of a nanomechanical oscillator. Nature 571, 537-540 (2019).

Ground-state phase diagram and universality of topological valence-bond-solid quantum phase transitions in a ferrimagnetic Heisenberg tetramerchain

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The ground-state ordering of a quantum mixed-spin Heisenberg tetramer chain composed of an alternate sequence of s=1 and S=3/2 dimers is studied in detail as a function of two considered exchange interactions ascribed to similar and dissimilar spin pairs. At zero magnetic field, the ferrimagnetic mixed spin-(1, 1, 3/2, 3/2) Heisenberg tetramer chain displays, depending on a mutual interplay between two considered exchange interactions, three distinct gapped valence-bond-solid phases separated by gap-closing quantum critical points. Using density-matrix renormalization group calculations we construct the full ground-state phase diagram as a function of the interaction ratio and magnetic field, which exhibits besides three gapped valence-bond-solid phases special Kosterlitz-Thouless and topological quantum critical points. A tangential finite-size scaling analysis is employed to obtain precise estimates of the zero-field valence-bond-solid transitions and unveil their common logarithmic correction to a power-law scaling of the correlation length.

Magnetic domains stabilization on curved substrate

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Magnetic domains and skyrmions have an excellent potential for application in new-generation spintronic devices. For instance, hexagonal arrays of skyrmions are good candidates for media recording [1]. It is demonstrated by the theory/simulations that bent surface topologies are suitable to stabilize the different magnetic textures, such as magnetic domain and skyrmion; moreover, vortex [2,3]. In this work investigated the magnetic domains in a self-supported micro-membrane using a self-organized array of polystyrene spheres (PS) templates. By colloidal lithography, we fabricated an array of polystyrene spheres to obtain a curved substrate and explore its effect. We produced substrates with different sphere diameters, which allow exploring the impact of curved nanocaps over the magnetic texture. Co/Pt and NiFe thin films (by magnetron sputtering) were grown over the PS arrays. The stacking, mainly the Co and NiFe layer thickness, was varied to obtain different magnetic properties. These systems allow us to explore magnetic texture in the function of the curvature and magnetic properties. By dissolution, we remove the polystyrene sphere to produce self-supported membranes with an "egg box" conformation. The structure and size of the membrane were investigated by scanning electron microscopyand transmission electron microscopy. We observed that all samples form self-supported membranes with a well-organized array of nanocaps. We investigated the magnetic domain on these membranes by magnetic force microscopy and electron holography. Tuning the curvature effect and magnetic properties of nanocaps, we obtained the hexagonal array of monodomain, stripes, vortex, or skyrmions. Our results show that it is possible to stabilize the different magnetic domains on self-supported nanocaps, which can be used for high-density recording information.

[1] ACS Appl. Mater. Interfaces 12 , 47, 53454(2020).
[2] Phys. Rev. B 104, 134427 (2021).
[3] da Fonseca, W. B. J. et al. Accepted.

Magnetic flux distribution in superconducting films decorated with an array of pinning centers: influence of the geometry of antidots

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Considering a noncentrosymmetric pinning texture composed of a square array of triangular holes, the magnetic flux penetration and expulsion are investigated experimentally and theoretically. A direct visualization of the magnetic landscape obtained by magneto-optical imaging on a Nb film is complemented by a multiscale numerical modeling. This combined approach allows the magnetic flux dynamics to be identified from the single flux quantum limit up to the macroscopic electromagnetic response. Within the theoretical framework provided by time-dependent Ginzburg-Landau simulations, an estimation of the in-plane current anisotropy is obtained and its dependence with the radius of curvature of hole vertices is addressed. Such simulations show that current crowding plays an important role in channeling flux motion, favoring hole-to-hole flux hopping rather than promoting interstitial flux displacement in between the holes. The resulting anisotropy of the critical current density gives rise to a distinct pattern of discontinuity lines for increasing and decreasing applied magnetic fields, in sharp contrast to the invariable patterns reported for centrosymmetric pinning potentials. This observation is partially accounted for by the rectification effect, as demonstrated by finite-element modeling. At low temperatures, where flux penetration is dominated by thermomagnetic instabilities, highly directional magnetic flux avalanches with a fingerlike shape are observed to propagate along the easy axis of the pinning potential. This morphology is reproduced by numerical simulations.

Acknowledgements to all coauthors of Ref. [1], whose contributions were vital for the complete understanding of the original results obtained in our laboratory at UFSCar.

Reference: [1] M. Motta, L. Burger, Lu Jiang, J. D. González Acosta, Ž. L. Jelic, F. Colauto, W. A. Ortiz, T. H. Johansen, M. V. Miloševic, C. Cirillo, C. Attanasio, Cun Xue, A. V. Silhanek, B. Vanderheyden, PRB 103, 224514 (2021).

Magnetic nanocomposite aiming to silica nano-spicules production for viral inactivation in 3D printing surfaces

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This work reports on the early stages of developing printable nanocomposites designed to inactivate viruses using silica nano-spicules involving magnetic nanoparticles (MNPs). This composite is formed of a photopolymerizable resin and a nanometric phase that, to be more hostile to viruses, must form artificial nano-spicules aiming at viral envelope damage. The methodology involved starts extracting magnetic nanoparticles from ferrofluids that were encapsulated with mesoporous silica in a star-shaped format (MNPs@SiO_2), as adapted from Zhang *et al.*(2013)[1]. These nano-spicules are expected to perforate the viral capsid and cause leakage of genetic material. The MNPs@SiO_2were dispersed in commercial photopolymerizable resins and printed in various formats using mask stereolithography (MSLA) printers with 405 nm LED sources. The printing parameters were evaluated to ensure the preservation of the 3D print quality. The samples were characterized using X-raydiffraction, atomic force microscopy, transmission, and scanning electron microscopy. Theresults showed that the nanocomposite was successfully prepared without affecting printability, and nanoparticles were attracted to the surface during printing. Finally, in collaboration with FIOCRUZ-PE (Aggeu Magalhães Institute), the antiviral action of the nanocomposites is being tested.

Acknowledgments

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) RIMA Project - Edital Capes 09/2020. and CNPq Brazilian Agencies.

References

[1] K. Zhang et al. J. Am. Chem. Soc. 135 (7), 2427-2430 (2013).

Magnetocaloric effect in compounds R_2Cu_2X (X=Cd,In).

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The magnetocaloric effect, which is the basis of solid-state refrigeration is the cooling or heating of magnetic materials upon magnetic field variation. The magnetocaloric effect is commonly characterized by the entropy change in an isothermal process (ΔS_{iso}) and by the temperature change (ΔT_{ad}) in an adiabatic process. Despite the bunch of experimental and theoretical works already published in the literature, a complete understanding of the physics behind the magnetocaloric effect is still lacking. The search for new magnetic materials with large values of the magnetocaloric quantities as well as new thermodynamic methods are some challenges in this area of research. In this paper, we discuss, from the theoretical point of view, the magnetocaloric effect in the series of compounds R_2Cu2X, where R stands for rare earth elements and X=Cd, In. For this purpose, we use a Heisenberg-like Hamiltonian with two magnetic sublattices and adopt the mean-field theory to treat the interactions between local magnetic moments. Our calculations show that the magnetocaloric quantities $\Delta S_{iso}$and ΔT_{ad}in the compounds Gd_2Cu_2Cd, Er_2Cu_2Cd, andTb_2Cu_2Cd show the usual behavior, i.e. they reach a sharp peak around the temperature ofthe phase transition and go down as the temperature moves from the critical region. On theother hand, in the compounds Dy_2Cu_2Cd Dy_2Cu_2In the curves of the magnetocaloric quantities exhibit two peaks which can be associated with two types of phase transitions. This fact, which is in good agreement with the available experimental data, is indeed due toan antiferromagnetic behavior observed in these compounds.

Permalloy nanowires growth by Haberland-type gas aggregation source

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Gas aggregation method is a widely employed technique to fabricate nanostructures, however, it faces several challenges to improve the output efficiency. One of them is the loss of species that occur within the aggregation chamber, mainly caused by entrapment zones. In the present work, Permalloy Ni80Fe20 nanostructures were grown on a silicon substrate by a Haberland-type gas aggregation source, following a singular synthesis strategy consisting in placing the substrates inside the aggregation chamber, with the idea of recovering lost species at the entrapment zone. The nanostructures obtained are made up of nano-chains grouped into microfibers with a certain preferential growth orientation and characterized by lengths of the order of micrometers and diameters between 500 and 20 nanometers. Furthermore, these nanostructures were characterized by X-ray diffraction, scanning electron microscopy, and magnetic response.

Skyrmion dynamics in the presence of defects inPd/Fe/Ir

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Magnetic skyrmions are noncollinear nanostructures which can be used as an information carrier in the design of new magnetic storage devices. Due to its topological feature, skyrmions can behave as particles, where they can be easily manipulated through the application of a polarized current density, for instance. Nonetheless, the current-induced motion of skyrmions has been experimentally investigated and different behavior for individual skyrmions were attributed to the presence of defects at the material [1]. Then, in order for these nanostructures to be realistically applied in new technologies, it is important to understand the skyrmions motion in the presence of inhomogeneities. In the literature, studies have been conducted regarding skyrmions’ dynamics with impurities, where gyrotropic motion, deflection and pinning behaviors were found according to attractive or repelling defects [2]. However, to the best of our knowledge, the effects of interfacial intermixing of atoms, in the skyrmions’ motion, has not been explored in the literature yet, specially a random distribution of defects. In this work, we use the Pd/Fe/Ir multilayer as our object of study to investigate skyrmions’ dynamics in the presence of intermixing of atoms at the interfaces Pd/Fe and Fe/Ir. From first-principles converged electronic structure results and magnetic interactions, the skyrmions’ motion is simulated through atomistic spin dynamics [3] with a polarized spin current density. Here, we consider several possibilities of intermixing, such as Pd and Ir atoms at the Fe layer, and analyze the type of motion as a function of the current density, initial position and percentage of defects.

References

[1] A. Hrabec et al., Nat. Commun. 8, 15765 (2017)
[2] I. L. Fernandes et al., J. Phys.: Condens. Matter 32, 425802 (2020)
[3] B. Skubic et al., J. Phys. Cond. Matt. 20, 315203 (2008)

Funding Acknowledgement: This work has been supported by FAPESP, CNPq, CAPES and eSSENCE.

Study of magnetic nanoparticles produced on hollow cathode magnetron sputtering gun

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The development of new methods for nanoparticles production has been one of the focal points in the recent years on material science research. This work consists of the development of a physical method for the production of magnetic nanoparticles (NPs). Here, we are using a new radial magnetron sputtering (RMS) gun, and the gas aggregation method. This RMS gun was developed and constructed in our lab and represents a improvement over the planar magnetron sputtering gun. In the setup, samples were prepared in the NP gun, installed as a new gun on a commercial magnetron sputtering system. Typically, the sputtering system operates at a pressure of 5 mTorr in the main chamber. However, inside the NP gun there is a pressure of 780 mTorr, under the Ar flux of 80 sccm. Atoms removed from the target are thermalized by the Ar flux and condensed to form NPs, which are extracted from the NP gun aerodynamically, flowing in direction to the substrate. The device is capable of producing films of NPs of different metals, including ferromagnetic metals, and also co-deposit the NPs simultaneously with other sputtered film. Cu and Co NPs were produced. For Cu NPs the averaged diameter was 5.79(8) nm and standard deviation of 2.97(5) nm, with crystallinity compatible with the FCC structure. Co NPs presented an average diameter of 3.88(3) nm and standard deviation of 1.74(2) nm, with crystallinity compatible with the FCC and HCP structures. When co-deposited with Si3N4 film, the agglomeration of NPs on substrate was minimized and Co NPs samples showed superparamagnetic behavior. This behavior was described by a combination of Langevin function and a distribution of diameters. Co NPs film dispersed with capping and buffer layer of Si3N4 showed average diameter of 3.1(1) nm and standard deviation of 1.2(1) nm. Co NPs film covered with Si3N4 showed average diameter of 3.8(2) nm and standard deviation of 2.2(2) nm. Both samples showed aging, with a loss of 66% of Co NPs, after one week.

Supercurrent diode effect and anisotropic vortex squeezing in non-centrosymmetric Rashba superconductors

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Most of 2D superconductors are of type II, i.e., they are penetrated by quantized vortices when exposed to out-of-plane magnetic fields. In a presence of a supercurrent, a Lorentz-like force acts on the vortices, leading to drift and dissipation. The current-induced vortex motion is impeded by pinning at defects. Usually, the pinning strength decreases upon any type of pair-breaking interaction perturbs a system.

In the talk I will discuss surprising experimental evidences showing an unexpected enhancement of pinning in synthetic Rashba 2D superconductors when applying an in-plane magnetic field. When rotating the in-plane component of the field with respect to the driving current, the vortex inductance turns out to be highly anisotropic. We explain this phenomenon as a direct manifestation of Lifshitz invariant that is allowed in the Ginzburg-Landau free energy when space-inversion and time-reversal symmetries are broken. As demonstrated in our experiment [Physical Review X 12 (4), 041020 (2022)], elliptic squeezing of vortices---an inherent property of the non-centrosymmetric superconducting condensate---provides an access to fundamentally new property of Rashba superconductors, and offers an entirely novel approach to vortex manipulation.

Another interesting feature of the non-centrosymmetric superconductors in the applied magnetic field is the supercurrent diode effect---the critical current in one direction exceeds its counterpart in the opposite one---what stems from the Cooper pairs with finite centre of mass momentum. In the pioneering experiment [Nature Nanotechnology 17 (1), 39 (2022)] we demonstrated the emergence of the supercurrent diode effect in the Josephson junctions based on synthetic Rashba superconductors made of Al-InAs quantum wells. In the talk, I will discuss novel experimental method---measurements of the Josephson inductance---and the semiquantitative microscopic model capturing all the essential features as observed in experiment.

Tailoring CeO2 Nanoparticles via Microwave-Assisted Hydrothermal Synthesis

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The influence of synthesis conditions on morphology and structure of CeO_2nanocrystals prepared by microwave-assisted hydrothermal method were studied by XRD, SEM and diffuse reflectance measurements. Decisive effect of the OH^-/Ce^{3+}molar ratio in the reaction mixture on the size and shape of CeO_2nanocrystals was established. Higher concentration of OH^-ions in the growth solution promotes bigger crystallite sizes as well as shape transformation from irregular (spheroidal) to needle-like and a mixture of needle-like and cubic nanoparticles. A difference in the energy gap was also observed, which can be attributed to differences in the oxygen vacancy concentration of the CeO_2nanoparticles, which is known to be influenced by the synthesis conditions [1]. Our results demonstrate that the MAHT method is an effective route to synthesize CeO_2nanoparticles with high crystalline quality and tailored morphology, where the molar ratio between precursors is crucial for controlling crystallite size and shape. These findings are valuable for the design and development of efficient catalysts, energy storage devices, and biomedical applications that require tailored magnetic properties.

The role of Tb, Dy and Ho intercalations on the superconducting and magnetic properties of $ZrTe_3$

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$ZrTe_3$ is a well-known monoclinic Van der Waals material with a highly anisotropic behavior emerging from a combination of quasi-one-dimensional electron-like sheets along with a three-dimensional hole-like sheet at the Fermi level. This combination of bands gives rise toa Charge Density Wave (CDW) ordering at 63 K, which coexists with superconductivity emerging at 2.0 K. The superconducting transition temperature (T_c) may be raised as the CDW is suppressed through atomic disorder, substitutional doping with selenium or intercalation with transition metals. In the latter, T_c more than doubles through intercalation of Cu, Ni or Ag with transition temperatures ranging from 3.8 to 4.6 K. Among the transition metals, Ni is the only magnetic intercalant that raises T_c, while intercalation of Fe was shown to be detrimental to superconductivity with the development of an antiferromagnetic order. Such diverse effect of the magnetic intercalant leaves a question open on what role the electrons of isolated inner shells may play on the properties of $ZrTe_3$, and the intercalation of elements with incomplete *f* electron shells may be a reliable way to study this aspect of the superconducting ground state. We successfully intercalated Terbium, Dysprosium and Holmium into $ZrTe_3$ single crystals with resulting transition temperatures in the vicinities of 5.3 K, along with a raise in susceptibility and signatures of antiferromagnetic order in all samples. The effects of the different rare-earth metals on the superconducting properties are compared by means of resistivity, magnetization and thermoelectric power measurements.

Acknowledgements

This project was financed by FAPESP under projects 2018/08819, 2021/14322-6, 2021/08966-8 and 2022/02691-0, CAPES under project 88887.595960/2020-00 and CNPq.

References

1 – Le J. Phys. Colloq. 44, C3 (1983) 2-5168.
2 – Chinese Phys. B 27, 087503 (2018).
3 – Phys. Rev. Lett. 106, 246404 (2011).

Tuning transport supercurrents in FIB-nanostructured Nb films by a magnetic field-induced weak-link to strong-link transition

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Superconducting weak links (WLs) give rise to exciting phenomena, paving the way for efficient electronics that integrate with the most advanced quantum technologies. Thus, patterning of superconducting films has become an essential process. However, introducing regions of suppressed superconductivity leads to a reduction of the device's critical current. In a previous report [1], we investigated the effects of a single groove-like WL created by Focused Ion Beam (FIB) milling in the properties of the normal and superconducting states of 180-nm-thick Nb films, revealing unexpected local peaks in their magnetization hysteresis loop. In this work [2], we explain such a phenomenon by exploring Magneto-Optical Imaging (MOI) to its full potential, locally quantifying magnetic and transport properties of the nanostructured Nb films. By mapping the magnetic flux distribution, we reveal that the local peaks are accompanied by a transformation of the sample's current domains. This process indicates a magnetic field-mediated increase in the WL critical current density (J_c^{WL}), quantitatively confirmed by MOI. To that effect, we reveal that the WL may behave as a strong link, allowing supercurrents to flow through it almost unaffectedly. By quantifying the flux inside and outside the film, we determine the vortex dynamics responsible for the J_c^{WL} enhancement and how it relates to the nanofabrication process. These results are corroborated by thermomagnetic model simulations. These effects bear a close resemblance to observations in high-temperature superconductors and may be used to enhance the performance of superconducting devices and in the development of new electronic applications.

The authors thank FAPESP (grant 2021/08781-8), CNPq, and CAPES.

[1] M.I. Valerio-Cuadros, D.A.D. Chaves, et al, Materials 14, 7274 (2021). Editor's Choice

[2] D.A.D. Chaves, et al, "Magnetic field-induced weak-link to strong-link transition in patterned superconducting films'' (In preparation).

Zero-field magnetic skyrmions in symmetric Pt/Co synthetic antiferromagnetic multilayers

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Magnetic skyrmions are topological solitons, emerging in the spin texture of magnetic systems without spatial inversion symmetry, such as non-centrossimetric crystals and multilayered thin films [1]. Skyrmion states exhibit a vortex-like spin structure with non-trivial topology, which gives the canted texture high stability and enable interesting spintronic applications. Skyrmion phases exist under narrow intervals of temperature and external magnetic field, hindering applications that require ambient conditions. Synthetic Antiferromagnetic Multilayers (SAF-MLs) are systems showing antiferromagnetic behavior, due to the antiparallel magnetization coupling of two ferromagnetic layers through a non-magnetic spacer layer, that have been reported as a skyrmion-hosting material in such conditions [2,3].

Here, we present experimental results of symmetric Pt/Co/Ru/Co/Pt multilayers, with varying Ru spacer thickness (t_{Ru}), by combining magnetometry measurements with magnetic force microscopy (MFM) imaging. The coupling energy was probed and correlated to the observed spin texture. Specifically, the hysteresis curves indicate three distinct types of SAF behavior for t_{Ru}= 0.4, 0.6, and 0.8 nm, exhibiting high saturation fields and very low remanence. Furthermore, MFM images indicates that the spin texture prior to magnetic field application is a mixed state containing worm-like stripes and skyrmions, while the remanent state texture reveals isolated skyrmions. These results show that skyrmions are present in compensated symmetric SAF–MLs under zero-field and room-temperature conditions, contributing to the realization of skyrmion-based spintronic devices.

Acknowledgements:

We thank funding agencies FAPESP (#2017/10581-1, #2020/07397-7), CNPq (#436573/2018-0), and CAPES (#88887.569085/202000).

References:

[1] Jiang, et al. Phys. Rep., 704, 1 (2017).

[2] Legrand, et al. Nat. Mater., 19, 34 (2020). [3] Duine, et al. Nat. Phys., 14, 217 (2018).

Ab initio study of Pd/Fe/Ir(111) bilayers decorated with Co nanostructures

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The study of skyrmions in recent decades has been of fundamental importance in the development of the next generation of information storage devices based on spintronics. However, before the complete implementation of this concept in technology, several challenges still need to be resolved. One such challenge is the Skyrmion Hall Effect, which results in the annihilation of the topological structure at the boundaries of the racetrack memory. Another problem is the difficulty in stabilizing skyrmions in the absence of a magnetic field. Experimental work conducted by J. Stephman et al. [1] shows that decorating the film edge of Pd/Fe/Ir(111) multilayers with Co/Fe patches can prevent skyrmion annihilation at the film edge and stabilize zero-field skyrmions and target states. In the present study, our aim is to provide theoretical support for the studied system. We investigate the electronic structure of different geometrical configurations of Co nanoislands in the Pd layer, with a focus on their magnetic properties. This study is conducted using first-principles calculations within the framework of the Density Functional Theory (DFT) [2], specifically utilizing the Real Space Linear Muffin-Tin Orbital in the Atomic Sphere Approximation (RS-LMTO-ASA) [3] within the Local Spin Density Approximation (LSDA). The use of a real space method presents advantages in treating systems with broken symmetry, such as the simulation of impurities and defects.

References

[1] J. Spethmann, E. Y. Vedmedenko, R. Wiesendanger, A. Kubetzka, and K. von Bergmann, Communications Physics 5, 19 (2022).
[2] P. Hohenberg and W. Kohn, Phys. Rev. 136, B864 (1964).
[3] S. Frota-Pessôa, Phys. Rev. B 46, 14570 (1992).

A DFT investigation on structural and electronic properties of the Ag-doped SrFeO3 material

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$SrFeO_3$(SFO) material is an oxide semiconductor with unpaired electrons on the Fe atoms as an advanced material in electronic properties and antiferromagnetic ordering. The SFO material is a cubic Perovskite with space group Pm-3m [1] with many possibilities of chemical modifications, for example, doping in Sr and Fe atoms, and presence of O vacancy [2]. The computational simulations were performed on DFT/HSE06 level in CRYSTAL17 software. The Sr, Fe, O, and Ag atoms were described by HAYWSC-311(d11f)G, 86-411d41G, 8-411d11G and 97-66314d(11) Gaussian-type basis sets, respectively. The $Ag_{0.25}Sr_{0.75}FeO_{2.875}$ (ASFO) material was simulated in a 2x2x2 supercell in two structures: i) the Ag doping on center and corner positions (ASFO-C); ii) the Ag doping on center and face positions (ASFO-F), including the O vacancy in both materials.

From the Ag doping in SFO structure, ASFO-C and ASFO-F models, showed distortions on the c lattice parameters. The $[FeO_6]$ octahedrons structures presented perturbations on the intermetallic angles (Fe-O-Fe) because of the modifications on the volumes, widely reported in literature as Breathing Distortions [3]. The SFO material presented a metallic electronic profile for conduction property, while the ASFO-C and ASFO-F materials, the Ag doping and the O vacancy, became the electronic profiles as half-metallic behavior, with bandgap of 1.70 eV in both materials. The SCDM showed distortions on the spin density of Fe atoms, and the phenomena of spin canting, denoting an anisotropic electronic behavior.

Acknowledgements:

The authors acknowledge the support of the CAPES and UEPG.

References:

ZWINKELS, M. F.M., et al., Catalysis Reviews Science and Engineering, vol.35, p.319-358 (1993).

SU, T. et al., International Journal of Hydrogen Energy, vol. 47, p. 16272-16282 (2022).

CAMMARATA, A.; RONDINELLI, J. M., Applied Physics Letters, v. 108, n. 21, p. 213109, 2016.

Anomalous Nernst Effect in a NiFe/Cr/NiFe magnetic trilayer

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Recent advances in the field of spintronics have stimulated interest in phenomena involving thermal and magnetic features, hence driving researches on their potential of application in green energy devices. Within this context, the Anomalous Nernst Effect (ANE) in nanostructured magnetic materials appears as a key phenomenon to optimally control and employ the internal energy dissipated in electronic devices, being dependent on, for instance, the magnetic anisotropy of the active element. In biphase magnetic systems, owing to the interplay of the distinct (hard/soft) magnetic properties, the dependence of theANE with the magnetization opens doors to quite-promising possibilities to manipulate the thermoelectric response by changing the magnetic anisotropies. Here we report an investigation of the structural features, magnetic properties and ANE in a NiFe/Cr/NiFe trilayer. We observe that the distinct structural properties of the bottom and top magnetic layers lead to the biphase magnetic behavior. We verify the ANE measurements have remarkable dependence with the anisotropies and magnetic response. Thermoelectric results seem to be very sensitive to the anisotropy of the biphase system than the magnetization ones, revealing interesting features of the magnetization process and placing place the ANE experiments as a very-sensitive tool to characterize anisotropic magnetic systems. In addition, the themoelectric experiments help us to understand the role of each layer in the ANE response for biphase systems, opening new perspectives to explore such systems for current and future technological applications.

Comparative Local Analysis of Ni/Ni3C Nanoparticles Synthed with Oleylamine/Oleic Acid and Oleylamine/Palm Kernel Oil Ligand Pairs: Structural and Magnetic Properties.

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In recent decades, Ni/Ni3C nanoparticles have been a topic of interest, especially for their catalytic and magnetic properties, promising as electrocatalysis, for example [1]. These systems are usually obtained by chemical methods, in the presence of ligands responsible for controlling their structure and morphology [2]. In this work, we carried out a comparative study of the hyperfine interactions occurring in Ni/Ni3C nanoparticles, synthed by thermal decomposition in the presence of oleylamine/oleic acid and oleylamine/palm kernel oil ligand pairs, at 513 K, for 3 hours. The local analysis was performed using perturbed correlation spectroscopy (PAC) in the temperature range from 30K to 300K, using the 111Cd probe, implanted in the samples by diffusion, during the synthesis. The parameters of the hyperfine interactions were compared to the results obtained by XRD, TEM and magnetization, indicating the obtainment of Ni/Ni3c nanoparticles of the core-shell type, showing a higher Ni3C content, greater distribution and lower saturation magnetization for nanoparticles synthed with palm kernel oil compared to those obtained with oleic acid. In addition, the hyperfine parameters showed the existence of regions with a quadrupole frequency of 24 MHz and a magnetic hyperfine field of 1T in both samples, which may be indicative of carbon-deficient Ni3C regions, predicted in the literature [3].

Acknowledgements: CAPES, for the financial support for the development of this research.

[1]Q. Qin and J. Hao, ACS Applied Materials Interfaces vol. 10, 17827 (2018)

[2] H. Wang and Y. Cao , ACS Applied Materials Interfaces vol. 9, 60 (2017)

[3] Z. Schaefer and K. Weeber, Chemistry of Materials vol. 23, 2475 (2011)

Composites based on Nitrogen-doped Graphene Aerogels and Magnetite Nanoparticles:Influence ofnitrogen on the magnetic and structural properties

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Graphene-based materials are known for their advanced properties, such as high area-to-mass ratio, electrical conductivity and capability to modulate their own properties through structural doping [1]. This capability makes these materials versatile and adjustable to current technological needs. Doping can also be adopted as an anchoring site for other nanostructures, which can be used for applications according to their nature [2]. In this context, nitrogen-doped graphene (NrGO) based aerogel were synthed through the hydrothermal method. NrGO were used as a spongy matrix for absorbing the aqueous suspension of magnetite nanoparticles (Fe_3O_4-NPs), forming the Fe_3O_4@NrGO composite. Also, Fe_3O_4@rGO was produced, used as a point of comparison to investigate the nitrogen doping effects on the structure of graphene and its influence on the magnetism of the composites. The Fe_3O_4-NPs showed superparamagnetic character (~5 nm by Scherrer equation). We expect that the NPs were anchored by physisorption in the NrGO structure, which presented an amount of 40.51 at% N-pyrrole by X-ray photoelectron spectroscopy (XPS). The composites were characterised by vibrating-sample magnetometry (VSM) and electron paramagnetic resonance (EPR) spectroscopy. The Fe_3O_4@NrGO presented saturation magnetization (M_S) of 0.7219 emu/g, more than twice higher than Fe_3O_4@rGO. We associate this effect to the expected higher hydrophilicity in NrGO, due to H-bonding interactions with nitrogen. The enhanced wettability should allow greater penetration of the Fe_3O_4-NPs suspension and greater incorporation of nanoparticles in NrGO. Low-field resonant peaks can be associated with the nitrogen paramagnetic centres of the NrGO, promoting efficient anchoring of Fe_3O_4-NPs.

Acknowledgements:
The authors gratefully acknowledge financial support from CNPq, CAPES and FACEPE.

References:

[1] S. J. LEE et al, Renewable Sustainable Energy Rev. 143, 110849 (2021)
[2] H. RASULI R. RASULI, J. Mater. Sci., 1-22 (2023)

Conventional s-wave superconductivity in non-centrosymmetric Re_3M (M = W, Ta, Nb) solid solutions.

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Weakly correlated, rhenium-rich superconducting compounds crystallize in a non-centrosymmetric structure and are potential candidates to host both singlet and triplet superconducting states. However, conventional BCS s-wave order parameter were reported in many of those materials, with some also breaking the time-reversal symmetry in the superconducting state. In this work, we study Re_3M (M = W, Nb, Ta) compounds that crystallize in the non-centrosymmetric cubic α-Mn (or A12) structure and have superconducting critical temperatures $T_c$ranging from ~ 4 to 9 K. Polycrystalline samples of Re_3M and its solid solutions, where one M is partially substituted for another, were synthed by arc-melting and heat treated subsequently at 1700 °C in vacuum. Motivated by the stability of the α-Mn structure in the Re-Ta and Re-Nb systems, we substituted partially Ta and Nb for W, leading to single phase NCS $Re_3W_{0.5}Ta_{0.5}$and $Re_3W_{0.5}Nb_{0.5}$even in as-casted samples, unlike the parent Re_3W compound. In what pertains to the superconducting properties, the low temperature heat capacity $C_p(T)$ data suggest conventional s-wave pairing in all samples [1], which is also supported by magnetization M(T), electrical resistivity $\rho(T)$, and thermal conductivity $\kappa(T)$ measurements. The estimated upper critical fields $H_{c2}(0)$, however, are sizable in comparison with the Pauli limiting field. These results are discussed within the context of the emergence of superconductivity in non-centrosymmetric materials.

Acknowledgements:
This material is based upon research supported by FAPESP (2013/07296-2, 2019/26141-6, 2022/02691-0, and 2022/10874-7), CNPq (Grant No. 301463/2019-0), and CAPES (Finance Code 001).

References:
[1] F. Abud, R.F. Jardim, N. Chaia and M.S. Torikachvili, Journal of Alloys and Compounds, (2023) doi:https://doi.org/10.1016/j.jallcom.2023.170259

Density Functional Theory and Perturbed Angular Correlation study of the AMnGe2O6 Clinopyroxeneseries

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Multiferroic materials have been under the spotlight due to their fundamental scientific interest and for potential applications in technology. Among these interesting materials are the group of compounds belonging to the Pyroxene family with general chemical formula AM(Si,Ge)$_2$O$_6$. More specifically, SrMnGe$_2$O$_6$[1]and CaMnGe$_2$O$_6$[2]are isostructural, crystallizing with monoclinic C2/c symmetry and are characterized by zigzag chains of MnO$_6$ octahedra linked by edge-sharing, separated by corner-sharing GeO$_4$ tetrahedra chains along the same axis. Due to this arrangement these systems present a rich diversity of low-dimensional magnetic properties.

In this work the structural and electronic properties of the CaMnGe$_2$O$_6$and SrMnGe$_2$O$_6$ clinopyroxene systems have been investigated by means of Perturbed Angular Correlation (PAC) measurements and Density Functional Theory (DFT) calculations using Quantum ESPRESSO [3], including a comparative analysis between the experimental electric field gradients and theoretical values obtained from DFT calculations. The enthalpies of formation of the constituent oxides of the broader AMnGe$_2$O$_6$(A = Be, Mg, Ca, Sr) series have also been examined through DFT calculations.

Acknowledgments:

The authors acknowledge project NECL under NORTE-01-0145-FEDER-022096, POCI-01-0145-FEDER-029454, and POCI-01-0145-FEDER-032527, and FCT through and CERN/FIS-PAR/0005/2017. R. M. gratefully thanks FCT-Portugal for his FCT grant. The work has been performed under the Project HPC-EUROPA3 (INFRAIA-2016-1-730897), with the support of the EC Research Innovation Action under the H2020 Programme; in particular, the author gratefully acknowledges the support of Alessandro Stroppa at CNR-SPIN and the computer resources and technical support provided by CINECA.

References:

[1] Lei Ding et al., J. Mater. Chem C, 2016, 4, 4236

[2] Lei Ding et al., Physical Review B 2016, 93 064423

[3] P. Giannozzi et al., J. Phys. Condens. Matter, vol. 29, no. 46, p. 465901, Oct. 2017

Determination of copper, zinc and lead from food supplements by restricted access magnetized carbon nanotubes followed by FAAS.

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Magnetic carbon nanotubes (Fe3O4-CNT) have advantages in determining metals via magnetic solid phase extraction systems, optimizing sample preparation. Surface modifications enhance absorptive capacity in complex matrices through protein mobilization, reducing competition for active sites. Restricted access materials (RAM) have shown great results for the determination of toxic metals in complex matrices [1]. This work objective is the use of Fe3O4-MWCNT coated with bovine serum albumin (BSA), for the determination of zinc, copper and lead in food supplements, by FAAS. The material was magnetized by addition of MWCNT to a suspension of Fe3O4 in ethanol, the reaction was maintained in inert atmosphere and stirring for 4 hours. For BSA immobilization, were used solutions of BSA in phosphate buffer, glutaraldehyde, and borohydride, which were separately added to the material, stirred, left to rest, and discarded, then, washed with ultrapure water. From ATR-FTIR spectrum, peaks at 1525 cm-1 and 1641 cm-1 indicates the presence of functional groups of BSA protein, as such, N-H, C-N (tyrosine) and N-H (amide). The BET isotherm showed that specific area was 105.3 g.m2, the pore volume and were 0.2119 cm3 and 10.51 nm, respectively, implying that the MWCNT modifications did not change textural properties, for this class of materials [2]. For quantification of zinc, copper and lead in aqueous standards solutions, the material showed a 34-fold gain in signal for the determination of Zn2+ (50µg. L-1, pH 8.0), while for Pb2+ (200µg. L-1, pH 6.0) and for Cu2+ (200µg.L-1, pH 8.0) the gain in signal were 20 and 16 times, respectively. Studies arebeing made to optimize the parameters that affect the magnetic solid phase extraction, to beapplied later in food supplements samples.

Acknowledgements:
To the funding agencies CAPES, UEL and CNPq.

Reference:
[1] A, Barbosa and V, Barbosa, Talanta 131, 213-220 (2015)
[2] P.M, Dos Santos and C.R.T, Tarley, JBCS 33, 37-47 (2022)

Development of scientific instrumentationfor characterization of magneto-optical properties of materials

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Using magneto-optical techniques for characterizing magnetic materials is a well-known approach which benefits from versatility and simplicity. For better exploring the potential of this technique, a data acquisition and signal processing program was developed for a magnetometer based on Magneto-Optical Kerr Effect (MOKE). This program was developed in the Labview language and it was used for interfacing our magnetometer and analyzing the acquired data. The program communicates with the system's instruments [1]: lock-in amplifiers, multimeters, current power supply, sensors, diode laser source (670nm) and others optical components. It's possible within the program to set signals profiles as input [2]: waveforms, maximum levels, resolutions and repetition cycles. As outputs, graphs of the results (hysteresis curve) and selectable numeric data, that can be exported as text formatto data analysis software. Through the configuration of optical components and program outputs, the user can characterize the magnetic properties of samples, characterizing some of its important features such as anisotropy, magnetic coercivity and remanence. The program provides programmable cycles of demagnetizations and it’s easily updated according to need and specific measurement/scientific goals. In addition, part of the existing system is already being used for time-resolved analyzes (TRMOKE) for the Kerr effect and also for the Faraday effect, covering, in this way, new possibilities of characterizing dynamical magnetic properties which is of interest for the development of devices allowing operation into the terahertz range.

References:

[1] D A Allwood et al 2003 J. Phys. D: Appl. Phys. 36 2175

[2] P T Squire et al 1988 J. Phys. E: Sci. Instrum. 21 1167

DFT quantum simulation on the magnetic states inthe SrFeO3 material

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The electronic superexchange described by Goodenough-Kanamori-Anderson (GKA model) [1] indicates that the magnetic coupling behavior between two magnetic sites intermediated by non-magnetic barrier as an O atom. In a Pm-3m cubic crystallographic arrangement, this material is a challenge to understand the GKA model. The HSE06 functional in DFT quantum simulations was performed from the CRYSTAL17. The HAYWSC-311(d11f)G, 86-411d41G and 8-411d11G gaussian basis sets descriptions were implemented for the Sr,Fe and O respectively. A 1x1x2 supercell built two [FeO6] magnetic clusters to investigateFM and AFM the magnetic ordering on the [Fe-O-Fe] connection under oxygen vacancy (Vo) perturbation. To evaluate the electronic profiles, the Electronic Band Structure (EBS) and Density of States (DOS) were discussed.

Structural data for the crystalline structure showed lattice parameters in agreement with experimental data in literature [2]. The magnetic ordering obtained to SFO structure simulated follows the literature, where is reported an AFM magnetic ordering to material. From the Vo perturbation, SFO:VO material, was noted that the Vo preserved the AFM ordering. The EBS presents a metallic behavior for the SFO material, and in the SFO:VO material, the Vo switch to a semiconductor material. Then, the overlap on between these electronic states shows an important interaction in intermetallic bond discussed as a molecular path for the GKA model, where SFO and SFO:Vo materials have a intense superexchange effect between Fe atoms an O non-magnetic atom acting like a bridge to interaction of two magnetic clusters.

Acknowledgements:
Acknowledgements to CAPES and UEPG by support.

References:

[1] GOODENOUGH, John B., Physical Review, vol.100, p.564 (1955).

[2] REEHYIS, M., et al, Physical Review B-Condensed Matter and Materials Physics, vol.85, p.1-15 (2012).

Effect of Iron Concentration on Magnetic, Structural, Morphological and Optical Properties of TiO2 Nanocrystals

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Diluted magnetic semiconductors (DMS) are a class of materials that exhibit unique magneto-optical properties that can be tuned by adjusting the dopant concentration. They are produced by incorporating magnetic ions, typically transition metal(TM) ions, into semiconductor hosts. The presence of these magnetic ions gives rise to DMS having a range of potential applications, including spintronics, magnetic sensors, and information storage[1-3]. Despite this, few studies in the literature have examined how the locations ofTM ions influence the structural and optical characteristics of titanium dioxide(TiO_2) nanocrystals. $TiO_2$is a semiconductor with ultraviolet absorption and emission,photostability,catalytic and osseointegration properties[4].In this study, the effect of iron ion concentration on the properties of TiO_2nanocrystals was investigated. The magnetic, structural, morphological and optical properties were analyzed using Magnetization, Electronic Resonance Paramagnetic, X-ray Diffraction, Raman Spectroscopy, Scanning Electron Microscopy and Optical Absorption Spectroscopy.XRD diffractograms and Raman spectra confirmed the formation of TiO_2 nanocrystals in the anatase phase.Additionally, the formation of other crystals was not observed. SEM images confirmed that the crystals were in the nanometer scale.In the AO spectra, the incorporation of Fe ions into the crystalline lattice of the TiO_2nanocrystals was verified by the variation of the band gap and the presence of characteristic absorption bands.To determine the locations where Fe ions were substituted for Ti ions, the Crystal Field Theory was utilized.Using the Tanabe-Sugano diagrams, it was demonstrated that Fe ions were integrated into tetrahedral and octahedral sites of the TiO_2NCs, depending on the doping concentration.The Mz and EPR results demonstrate the presence of Fe ions and how the concentration altered the magnetic properties.

Effect of reaction on the microstructure and superconductivity in Bi/Ni Nanoparticle/Thin FilmSystems

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Superconductivity in Bi-Ni bi-layer system attracted great attention because both Ni and crystalline Bi are non-superconducting above 50 mK and no agreement has been made for the interpretation. We studied the bilayers of Bi-Ni and concluded that the superconductivity is from the formation of the $NiBi_3$or NiBi intermetallic compounds. Atom diffusion is highly involved in the reaction of Ni and Bi [1,2]. To give more evidence to this conclusion, we studied hybrid systems with Bi thin film (TF) layer and Ni nanoparticles (NPs) on top, or Ni layers with Bi nanoparticles on top. The NPs of Ni or Bi are formed before depositing on the layers and the activity of the atoms in NPs is much smaller than the single atoms. In this case the diffusion would be more difficult to occur. The TEM images and the diffraction patterns of the hybrid samples indicate that Ni reacted with Bi in all samples and formed the intermetallic compounds $NiBi_3$. Pure Bi and Ni remains in samples of Ni NPs deposited on Bi. On the other hand, the Ni TF deposited on the Bi NPs does not show the existence of pure Ni. Electrical transport measurements indicate that Ni TF samples deposited on Bi TF shows the narrowest and most complete superconducting transitio the Ni NP samples deposited in the Bi TF, however, shows the widest and most incomplete superconducting transition due to more difficulty of reaction when depositing NPs of Ni atoms on Bi. The reaction of Bi and Ni induced the agglomeration of Bi and generated inhomogeneity in the thickness of the samples. The results strongly support the conclusion that superconductivity is from the intermetallic $NiBi_3$formed by the reaction of Ni and Bi.

Acknowledgments: The authors gratefully acknowledge FAPERJ and CNPq for the support.

References:
[1] Liying Liu, et al., Phys. Rev. Materials 2, 014601 (2018).
[2] Liying Liu et al., Mater. Chem. and Phys. 260, 124112, (2021)

Effects of structure and morphology on the magnetism and magnetic fluid hyperthermia response of gold–iron oxide (Au/Fe_3O_4) heterostructures

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Noble metal nano-heterostructures in combination with iron oxide have aroused great interest due to their multiple modifiable surfaces, unique magnetic properties and biocompatibility, making them promising for effective biomedical applications such as magnetic fluid hyperthermia (MFH). MFH selectively destroys or weakeni malignant tumor cells by increasing their temperature above a critical value. However, a challenge to be overcome is the limited heating capacity or low specific absorption rate (SPA) of the NPs. To improve the heating efficiency, researchers have employed strategies such as enhancing anisotropy, varying volume, developing core-shell structures, among others. In this work, we will present a study of the structural, morphological, and magnetic characterization of Au/Fe304 hybrid NPs and their corresponding ferrofluids explored for MFH applications. The influence of the gold core and the magnetic response of the magnetic shell will be systematically studied. Although many studies have been carried out with iron oxide NPs, the potential of the Au/Fe304 combination has not been systematically explored. The Au/Fe304 NPs were prepared by thermal decomposition of iron precursors in the presence of pre-formed ~7 nm Au seeds. The crystal structure and morphology of the NPs were determined by X-ray Diffraction (XRD), Transmission Electron Microscopy (TEM), and magnetic properties were determined by magnetization measurements. The SPA was obtained for different NPs concentrations dispersions and in different rheological conditions (i.e., different fluid viscosities), with frequencies between 100 kHz and 800 kHz and magnetic field amplitude of up to 56 mT. The resulting SPA values were analyzed in terms of the different possible mechanisms that determine the magnetic losses. This review will provide information on structure control in tuning the function of heterogeneous or hybrid materials in the study of magnetic fluid hyperthermia.

Enhanced spin Seebeck effect in the paramagneticphase of the three-dimensional Heisenberg antiferromagnet $RbMnF_3$

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$RbMnF_3$ is an insulating antiferromagnet with a perovskite crystal structure and Néel temperature $T_N = 83$ K. Having magnetic ions with a S electronic ground state in a cubic arrangement, its magnetic anisotropy vanishes so that it is a nearly perfect three-dimensional Heisenberg antiferromagnet. Here, we report measurements of the spin Seebeck effect (SSE) in bulk samples of $RbMnF_3$ with the shape of a slab onto which a thin Pt film strip is deposited. By applying a thermal gradient perpendicular to the slab plane, we detect an electric voltage along the Pt strip due to the charge current resulting from the conversion of the thermally generated spin current by means of the inverse spin Hall effect. By varying the magnetic field applied on the plane of the slab, we observe the antisymmetric step variation typical of the SSE both in the spin-flop and paramagnetic phases. Surprisingly the SSE increases with temperature in the paramagnetic phase and becomes comparable to the one in the ordered phase. This enhancement is attributed to a combination of the free spin fluctuations of paramagnetic insulators with the thermal conduction of long-lived paramagnons made possible by the short-range order that persists above T_N.

Estimate of weight-to-power ratio of a Tesla-type thermomagnetic motor

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Thermomagnetic motors (TMs) are a class of devices focused on obtaining mechanical work exploiting the magnetic phase transition of a working material (WM), when submitted to a temperature change around its Curie temperature (Tc). An important factor of such devices is the weight-to-power generation ratio. Seeking to estimate such ratio, an interpolation study of the theoretical results presented in [1] was made for a Tesla-type TM, which undergoes a magnetic Brayton cycle (MBC). Using the data of specific work per cycle from [1], two scenarios were considered: (a) the mass of WM required to produce 1kW of mechanical power; (b) the power produced with 1kg of WM. This was investigated for different conditions of temperature around Tc. It was considered an uniform temperature distribution along the WM and an applied magnetic field change of 1.5 T. Demagnetization effects were ignored and the cycle times were imposed. Two materials, with first and second order magnetic transition, respectively, were considered as WMs: MnAs and Gd. In (a), smaller cycle times required less mass of WM to generate 1kW. Gd requires much more mass and has a larger mass span over the values of ΔT when compared with MnAs. In (b), as expected, for the same value of mass, MnAs generates greater values of power then Gd. It is important to highlight that the permanent magnets mass was not taken into account. However, as a first estimate, these results can be used to get an order of magnitude of the size and weight-to-power ratio of TM undergoing a MBC.

Acknowledgements:

The authors would like to thank the National Agency of Petroleum, Natural Gas and Biofuels (ANP) for its support in the development of this work.

References:

[1] C.V.X. Bessa, L.D.R. Ferreira, O. Horikawa, J.C.B. Monteiro, F.G. Gandra, S. Gama. On the influence of thermal hysteresis on the performance of thermomagnetic motors. J. Appl. Phys. 122 (2017) 244502.

Extraction of macronutrients from microalgae using cellulases immobilized on nanomagnetic supports

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Microalgae biofuel is of great promise, but extracting their macronutrients is challenging given the thickness of their cell walls [1]. The enzymatic hydrolysis of cellulose can break them down, but this process is unprofitable on account of the enzyme' s poor stability and efficiency [1,2]. A practical and efficient solution to improve its stability is the immobilization of cellulase on magnetic nanoparticles (MNPs), which also allows for its recovery and reutilization [1,2]. Thus, this work sought to test the synthesis and functionalization of MNPs and the immobilization of cellulase on their surface. The MNPs were synthed by reducing iron salts in an alkaline medium, coated with tetraethyl orthosilicate (TEOS), and functionalized with (3-aminopropyl)triethoxysilane (APTS) and (3-mercaptopropyl)trimethoxysilane (MPTS). They were then characterized by transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, X-ray diffraction (XRD), and UV-Visible absorption spectroscopy, showing strong magnetization [3]. Once resuspended in a cellulase solution, the bioconjugation assay results revealed that the MNPs coated with TEOS, functionalized with APTS, and activated with glutaraldehyde showed the highest rate of cellulase immobilization, achieving a recovery of 250 mg/g of MNPs in just 1 hour.

Acknowledgements:

The Espírito Santo State Foundation of Research and Innovation (FAPES), the Federal University of Espírito Santo (UFES) and to the Federal Institute of Espírito Santo (IFES).

References:

[1] HOSSEINI, S. H. *et al.* **Journal Of Agricultural And Food Chemistry**, v. 66, n. 4, p. 789-798 (2018).

[2] RAJNISH, K. N. *et al.* **International Journal Of Biological Macromolecules**, v. 182, p. 1793-1802 (2021).

[3] SILVA, T. A. *et al.* **Scientific Reports**, v. 12, n. 1 (2022).

Fe_3O_4 magnetic nanoparticles for Magnetic Fluid Hyperthermia

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Magnetic hyperthermia is the conversion method of electromagnetic field energy into heat through magnetic nanoparticles (MNPs), i.e., when an external AC magnetic field (RF range) is applied to the nanoparticle magnetic colloids, energy in the form of heat is dissipated into the medium. Quantification of this heating can be obtained from the specific absorption rate (SAR), from temperature increase as function of time when the RF field is applied in the colloidal system. In this work, we studied iron oxide magnetic nanoparticles (IONPs), specifically Fe3O4 (magnetite), with diameters between 6-25 nm synthetized by high-temperature thermal decomposition of organometallic precursors. The crystal structure and morphology of these IONPs were determined by X-ray Diffraction (XRD) and Transmission Electron Microscopy (TEM). The magnetic properties were obtained by DC magnetic measurements using SQUID magnetometry. By controlling the size of these samples, we were able to compare (between the different sizes of nanoparticles) the SAR in magnetic hyperthermia experiments for frequencies between 90 kHz and 300 kHz and magnetic field amplitude up to 56 mT. The measured SAR value of the 17 nm MNP dispersed in paraffin presented an interesting value of 804 W/g in 103 kHz and 56 mT configuration that does not correspond to the linear response theory, where it was expected to achieve the maximum SAR value of 635 W/g. This sample presented a saturation magnetization of 80 emu/g and coercivity fields of 0.397 kOe at 2 K and 0.064 kOe at 300 K.

Induction hysteresis loop tracer with μ-emu sensitivity

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Standard experimental tools for probing the magnetic moment of materials research include the superconducting quantum interference device (SQUID) and vibrating sample magnetometer (VSM).

A less common alternative is the use of an ***induction magnetometer*** or equivalently a ***magnetic flux-meters***, which can get magnetization directly by integration of Faraday''s law induction voltage.

Despite this alternative is by far more affordable than SQUID and VSM devices and intrinsically more versatile, its usage for the study of magnetic thin films is up to now, not possible, due to its smaller sensibility in the range of 10^{-4}emu.

In this work, we present a table-top, own design, very low-cost hysteresis loop (H-loop) tracer based on magnetic-flux quantification, suitable for the study of ferromagnetic thin films. Our device is capable to obtain H-loop curves of ultra-thin films of permalloy (NiFe), in a matter of minutes (Fig. 1). having a verified sensitivity limit in the order of $\sim$ 10$^-6$ emu.

In addition, our device is especially suited for the study of soft magnetic materials, being capable to detect changes in the coercive field as small as 0.1 Oe. The performance of our device was assessed by the study of the magnetic moment per unit area in NiFe ultra-thin films deposited on silicon dioxide substrates, where a native oxide layer on NiFe with a thickness equal to 1.6 $\pm$ 0.2 nm was determined.

Investigation of superconductivity which emerges fromRu intercalation in a Dirac semimetal of $ZrTe_2$ composition

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Dichalcogenide materials are characterized by the chemical formula MX_2, which structure is defined by X-M-X type layers, that weakly interact with each other by Van der Waals type interaction [1]. The $ZrTe_2$ compound is a transition metal dichalcogenide which, according to the modern view of electronic structures, has a non-trivial topology and can be classified asa Dirac semimetal. The present study aims at the controlled intercalation of Ru between Te atoms in the $ZrTe_2$ compound with the purpose of obtaining a new topological superconducting material, since a relatively extensive range of atoms and molecules, when intercalated between the layers of these materials, is able to suppress the charge density wave transition, inducing superconductivity [2], two competitive phenomena to the Fermi surface, which, in general, coexist in this class of materials. The study was carried out by growing high-quality single crystals using the isothermal chemical vapor transport method [3], which were characterized by X-ray diffraction and energy dispersive spectroscopy, and the four-probe method was used for resistive characterization. So far, single crystals of nominal $Ru_{0.1}ZrTe_2$ composition have been synthesized and characterized, in which the phenomenon of multiband superconductivity was successfully identified, with critical temperature of 3.0 K. The occurrence of this behavior in this compound was associated with a possible hybridization between the p orbitals of Te and the d orbitals of Ru, which wouldbe responsible for originating a chemical pressure in the matrix.

Acknowledgements:

This project was financed by FAPESP under project 2022/12392-0.

References:

[1] E. Gibney; Nature 522, 274 - 276 (2015)

[2] L. Fang, Y. Wang, P. Y. Zou, L. Tang, Z. Xu, H. Chen, C. Dong, L. Shan and H. H. We Physical Review B 72, 014534 (2005)

[3] L. E. Corrêa, L. R. de Faria, R. S. Cardoso, N. Chaia, M. S. da Luz, M. S. Torikachvilli, A. J. S. Machado, Journal of Crystal Growth 595, 126819 (2022)

Investigation of the adsorption of the tetracycline antibiotic by NiFe2O4 nanoparticles

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Antibiotics are essential medicines for the prevention and treatment of bacterial infections in humans and animals. The presence of this antibiotic in the aquatic environment can cause toxic effects and potential development of resistant bacteria [1]. In this work, nickel ferrite (NiFe2O4) nanoparticles (NPs) were synthesized by coprecipitation, annealed at 623 K and characterized by X-ray diffractometry (XRD), Fourier transform infrared spectroscopy (FTIR), Mössbauer spectroscopy (ME), vibrating sample magnetometry (VSM), electrophoretic light scattering and gas adsorption. In addition, the application of NiFe2O4 NPs as an adsorbent for the antibiotic tetracycline (TCN) was investigated. The experimental data confirmed the formation of the partially inverse spinel phase of NiFe2O4 with crystallite sizes of 6 nm, superparamagnetic behavior, low saturation magnetization and coercivity, isoelectric point close to pH 7 and mesoporous structure. The non-linear equilibrium data better fit the Redlich-Peterson model, suggesting that the isotherm form approximates that of Freundlich. The kinetics non-linear fit indicated that the experimental data better fit the Elovich model. In addition, FTIR showed a shift from 1580 cm-1 to 1595 cm-1 in the absorption band v(C=O). This perturbation may be related to the formation of a hydrogen bond between the hydroxyl groups on the NiFe2O4 surface and the C=O group of the TCN keto-enol function.

Acknowledgements:
CNEN, FAPEMIG, CNPq.

References:

[1] REDDY, D. H. K. and YUN, Y.-S, Coordination Chemistry Reviews vol. 315, page 90-111 (2016)

Logic operations in Y-shaped spin chains

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In the present work, we investigate the achievement of logic operations on Y-shaped spin wave logic gates, composed of two input chains coupled to an output chain. We analytically calculated the transmission spectrum for a ferromagnetic and antiferromagnetic Y-shaped chains. We obtain the logical operations AND, OR and XOR, and demonstrate that the desired logical operation can be controlled by manipulating the phase difference between the incident waves. A study of the contrast ratio was also used to better characterize the efficiency of the logic gate.

References:

M. Cieplak and G. Ismail. Localised spin waves in disordered ferromagnetic chains. J. Phys. C: Solid State Phys. 20 1309, 1987.

K. Wang, S. Hu, F. Gao, M. Wang, D. Wang. Dual function spin-wave logic gates based on electric field control magnetic anisotropy boundary. Appl. Phys. Lett. 120 (14): 142405, 2022.

Magnetic and magnetocaloric properties in $Dy_{1-x}Tb_xAl_2$ with x = 0, 0.15, 0.35 and 0.4

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In this work, we compare the magnetic and magnetocaloric properties of compounds with $Dy_{1-x}Tb_xAl_2$(where x = 0, 0.15, 0.35, and 0.4) modeled using a Hamiltonian that includes exchange interactions among Dy-Dy, Dy-Tb, and Tb-Tb ions, as well as the crystalline electric field and the Zeeman effects, with experimental results.To reproduce the experimentally observed ferromagnetic ordering and spin reorientation transition temperatures as the Tb concentration (x) varies [1], the exchange interactions within the rare earth sub-lattice were treated as free parameters and adjusted to match the experimental results.Our findings indicate that the heat capacity of polycrystalline materials in non-zero magnetic fields can be satisfactorily reproduced by averaging the main three field directions with respect to the crystallographic coordinate system. Moreover, we demonstrate a reasonably good agreement between the experimentally [1] determined and theoretically predicted magnetocaloric effects.

References:
[1] M. Khan, et al., Journal of Materials Chemistry C, vol. 5, p. 896, 2017. DOI: 10.1039/c6tc05384j

Magnetic characterization of composites of graphitic carbon nitride with magnetite

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Since the discovery of graphene in 2004, this material has been widely studied due to large range of applications associated with its electronic structure, a band gap frequency inside the visible spectrum, high chemical stability, high thermic resistance, biocompatibility, high electrical conductivity and hardness. Others carbon base materials has been explored, among them the graphitic carbon nitride (g-C3N4) that presents various morphologies, likeporous nanosheets, nanorods, nanoflakes and nanotube. In particular, the combinations of g-C3N4 with magnetic materials to improve photocatalytic activity, pharmaceuticals nanocarriers action and hydrogen generation has been largely search, among the well-known and study magnetic materials used, are ferrite and magnetite (Fe3O4). Until this moment has not been find, any work that aims at the enhancement of magnetic properties on composites of g-C3N4/ Fe3O4. The present work uses urea synthesis to provide a most graphitic state to graphitic carbon nitride, which allows an easier incorporation of magnetite in to the material. The composites of (1-x) g-C3N4/ (x) Fe3O4 with x= 0,01; 0,05; 0,10; 0,20; 0,30; 0,40 has their structure analysed with DRX and MEV techniques and the magnetic properties were characterized with VSM magnetometer. The results show that the formation of Fe3O4 clusters induced a superparamagnetic behavior in samples that depend on synthesis route and the distribution of grains in g-C3N4 matrix.

Magnetic field driven superconductivity in Nb-Bi composites

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We report the results of magnetoresistance and magnetization measurements performed on granular Nb-Bi composites prepared under different pressures (P) up to P = 2 GPa, where Nb is conventional s-wave superconductor (SC) and Bi is the elemental semimetal Bismuth. One of the characteristic physical properties of conventional superconductors is the suppression of superconductivity by applied magnetic field. However, the main result of the present work is the observation of superconductivity in Nb-Bi granular composites through the application of the external magnetic field. We discuss our finding within a framework of theoretical models for the negative intergranular Josephson coupling, as well as the possibility of emerging Majorana fermions at the Bi/Nb interfaces. Our recent observation of the pressure-induced ferromagnetism in Bismuth points out on the interrelation between the ferromagnetism and field-induced superconductivity in Nb-Bi.

Magnetic properties of superparamagnetic $Mn_{0.8}Zn_{0.2}Fe_{2-x}Ce_xO_4$ synthesized using the sol-gel auto- combustion route

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Cubic ferrites are metal oxides with composition AB_2O_4, where A and B are divalent and trivalent cations, respectively, and one of the trivalent cations is the Fe^{3+} ion. There are two different sites in its crystal structure, tetrahedral sites, where the cations are surrounded by four oxygen ions, and octahedral sites, where the cations are bonded to six oxygen ions. The cation distribution among the tetrahedral and octahedral sites is partially responsible for the magnetic properties of a cubic ferrite. Recently, rare earths have been explored as cation substitute; the results suggest that introduction of small concentrations of them leads to a weaker magnetic response, which could be desirable for applications such as magnetic hyperthermia [1]. In this study, we investigated the influence of Ce^{3+}substitution on the magnetic properties of the manganese-zinc ferrite $Mn_{0.8}Zn_{0.2}Fe_{2-x}Ce_xO_4$ with x = 0; 0.015; 0.030; 0.050 and 0.100. The samples were synthesized by the sol-gel auto-combustion method and characterized by X-ray diffraction (XRD), Mössbauer spectroscopy (MS), Raman spectroscopy (RS), transmission electron microscopy (TEM), electron diffraction by selected area diffraction (SAD), and vibrating sample magnetometry (VSM). The XRD results show that the crystallite size are less than 8 nm for all samples and the cubic ferrite phase has obtained. The room temperature MS spectra show doublets that suggest superparamagnetic behavior and MS spectra at 5 K show two sextets, indicating the presence of Fe^{3+}in tetrahedral and octahedral sites. The TEM and SAD results confirm the small size and the crystal structure of the particles, respectively, and VSM results show a decrease in the saturation magnetization when cerium atoms are incorporated in the material.

Acknowledgments: The authors thanks CBPF, CAPES, CNPq, FAPERJ and FAPEMIG for financial and experimental help.

References:

[1] Shaw et al, Journal of Magnetism and Magnetic Materials 434, 181-186 (2017)

Magnon heat-current approach for the spin Seebeckand spin Peltier effects based on the Boltzmann's theory

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Spincaloritronics essentially deals with phenomena that exhibit a connection between spin and heat currents. In this field, two effects have been investigated (experimentally and theoretically) by several research groups: the spin Seebeck effect (SSE), and the spin Peltier effect (SPE). In SSE, a temperature gradient applied to a magnetic material (MM) gives rise to a spin current in it, which can be measured with the aid of a metallic layer (ML) due to the inverse spin Hall effect [1]. In contrast, the reciprocal effect, the SPE, a temperature variation at the interface of the MM/ML system can be verified when a charge current flows in the ML layer. As a consequence of the spin Hall effect, the charge current converts into a spin current, and the spin flux through the interface causes a temperature variation in this region [2].

In this work, a unified theoretical approach is proposed for both effects, which is based on Boltzmann''s theory in the relaxation time approximation. This allows the definition of the magnon spin and heat currents. The model is developed for an insulating ferromagnetic material. The treatment is carried out considering the magnonic heat current and its corresponding continuity equation. The connection between the spin and heat currents at the interface is performed by assuming that each magnon carries angular momentum and an average energy, the last defined from a occupation distribution close to equilibrium [3]. As a consequence, a unique dependence on the thickness of the magnetic layer is directly obtained for both effects. Finally, our calculations are compared with experimental data reported in the literature by other groups for the YIG/Pt.

References:

[1] T. Kikkawa, K. Uchida, S. Daimon, Z. Qiu, Y. Shiomi and E. Saitoh, Physical Review B, 92, 064413, 2015.

[2] S. Daimon, R. Iguchi, T. Hioki, E. Saitoh and Ken-ichi Uchida, Nature Communications, 7, 13754, 2016.

[3] S. Zhang and S. Zhang, Physical Review B, 86, 214424, 2012.

Manipulating Magneto-Optical and Interface Effects in Nanostructures

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The recent interest of the scientific community in magneto-optical interactions and interface effects has opened the possibility of a global understanding of characteristics not yet dazzled in materials. In this context, nanowires and films of magnetic materials have always been sources of research. Based on this interest, we studied both magneto-optical interactions and interface effects in ferromagnetic nanowires and antiferromagnetic thin films [1-3]. In the results involving nanowires, we observed that the intensity value obtained from the interactions provides information about their magneto-optical behavior, in addition to obtaining the quantitative value of these interactions, which are magnetizing and demagnetizing [1,2]. On the other hand, our experiments at paramagnetic/antiferromagnetic interfaces show the existence of a new magnetic effect detected by ferromagnetic resonance with and without applied electric current [3].

Acknowledgements: This research was supported by Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Financiadora de Estudos e Projetos (FINEP), and Fundação de Amparo àCiência e Tecnologia do Estado de Pernambuco (FACEPE).

References:

[1] Silva, W. W. G., Degiorgi, S. V. B., Holanda, J., Experimental Observation of a Magnetic Interfacial Effect. Adv. Mater. Interfaces , 10, 2202143 (2023). https://doi.org/10.1002/admi.202202143.

[2] Silva, W.W.G., Dias, A.d.A., Rodrigues, A.R. et al. An experimental analysis from the magnetic interactions in nanowire arrays. Appl. Nanosci. (2023). https://doi.org/10.1007/s13204-023-02769-7.

[3] Silva, W.W.G., Hildever, L.M.C.S., Santos, M.C.G. et al. Analyzing the Magnetic Influence on Magneto-optical Interactions. J. Supercond. Nov. Magn. (2023). https://doi.org/10.1007/s10948-023-06530-7.

Microwave assisted hydrothermal synthesis andmagnetic properties of 50(Na0.5Bi0.5)TiO3 - Ba0.06TiO3/50CoFe2O4 nanopowders

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Heterostructured multifunctional materials have been extensively studied due to their physical properties and potential in numerous applications. However, obtaining these materials is challenging. New synthesis protocols need to be developed to ensure adequate homogeneity between the constituent phases. Microwave-assisted hydrothermal synthesis offers advantages such as homogeneous composition, low temperatures, and shorter synthesis time compared to other methods. Therefore, this work aimed to prepare the 50(Na0.5Bi0.5)TiO3 - Ba0.06TiO3/50CoFe2O4 (NBT-BT6/CFO) magnetic system, using the microwave-assisted hydrothermal to achieve a particulate magnetic nanopowders with a consistent distribution of phases and reduced energy consumption during the synthesis process. The nanopowders were synthed in two stages: first, the NBT and BT phases were prepared at 200 °C for 2 hours, and successively the CFO phase was prepared at different temperatures (120-150 °C) and times (15 to 45 min) in the one pot. From the results of the morphological analysis by SEM and TEM and structural analysis (XRD), the formation of the NBT, BT, and CFO phases were observed on a nanometric scale and homogeneous distribution of the particles. In addition, the nanopowders presented a saturation magnetization of around 39.8 emu/g with a magnetic hysteresis that can be related to soft magnetic materials. Thus, one-pot microwave-assisted hydrothermal synthesis of multiphase nanoparticulate powders has potential to be used in the preparation of nanopowders with magnetic applications.

Acknowledgements: The authors would like to thank FAPESP process n. 2017/13769-1, CNPq process n. 431304/2016-5, CAPES process 88887.514769/2020-00 for financial support and scholarship. This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Molecularly imprinted magnetic polymer using semi-covalent chemical imprinting approach for application preconcentration of oxytetracycline and determination for HPLC-DAD.

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Magnetic dispersive solid phase extraction employing magnetic molecularly imprinted polymers has been utilized due to the selective adsorption of the target by the polymer1. The present work had as objective to develop a new analytical method based on preconcentration of oxytetracycline (OTC) using a magnetic molecularly imprinted polymer (Fe3O4/SiO2/MIP). The innovative feature of this work relies upon semi-covalent synthesis, in which the template-monomer complex was synthesized by the reaction of the functional monomer 3-triethoxysilylpropyl isocyanate (ICPTES) via covalent bond with the OTC molecule. Removal of the OTC by chemical cleavage produced specific OTC recognition sites. The non imprinted polymer was synthesized by replacing the OTC-ICPTES complex for the (3-aminopropyl)triethoxysilane with the goal of obtaining the same functional groups of the magnetic-MIP. A specific superficial area of 216.0 m2g-1 was observed for Fe3O4/SiO2/MIP superior to Fe3O4/SiO2/NIP (103.6 m2g-1). From competitive adsorption studies it was observed that the Fe3O4/SiO2/MIP presented greater selectivity to OTC compared to the concomitants and comparison to Fe3O4/SiO2/NIP. The maximum adsorption capacity of Fe3O4/SiO2/MIP was 8.78 mg g-1. The result is considered satisfactory, especially due to the reduced extraction time (80 s). The method is based on the preconcentration of 40.0 mL of standard/sample (pH 4.0) vortex-assisted with 10 mg of Fe3O4/SiO2/MIP during 60 s. The elution was performed with 400 μL of oxalic acid 0.01 molL-1/acetonitrile (77.5/22.5 v/v). The method presented limit of quantification and detection of 0.26 and 0.89 μg L-1, respectively, and preconcentration factor of 90.44 times. The method was applied to determine OTC in water and milk samples with recovery resultsranging from 94.0-99.0%.

Acknowledgements: UEL and CAPES.

References: [1] CHEN, H. et al. Bio-Inspired Imprinting Materials for Biomedical Applications. Advanced science, v. 9, p. 1-38, 2022.

Net-Shape Nd-Fe-B Permanent Magnets made by Pressure-Less Process

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Nd-Fe-B sintered magnets are experiencing significant growth in demand, mostly due to an increase in greener applications such as electric motors in transportation. Nd-Fe-B sintered magnets are almost exclusively produced via the traditional Powder Metallurgical Route, which includes a pressing step before the final sintering operation. However, the necessary fine-grained microstrostructure after sintering leads to the use of a very fine metallic powder, not ideal for pressing, due to the lack of flowability and consequently, density gradients in the green body. Such gradients cause undesired distortions in the sintered part, which must be corrected by machining operations and consequently generate large material losses. The highly oxidized swarf can not be directly reused, and value-added elements (rare earths - e.g. Nd, Pr, Dy) must be recycled by energy and chemical intensive processes, attenuating the benefits of the magnets for a greener economy. A promising process approach is presented in this work that makes use of fine alloy powder, produced by autogenous milling, which can be sintered to full density without the pressing stage (Press Less Process, or PLP [1]). In this work, the powder was fed into graphite and titanium molds in different amounts in order to vary the apparent density, followed by a special vibrating procedure and a pulsed magnetic field that was used to develop the crystalline texture. The suitably packed molds were then loaded into a tubular furnace for the sintering stage (sintering at 1050 ℃/1 hour followed by heat treatments at 900 ℃/1 hour and 600 ℃/1hour). The magnetic properties of the magnets obtained by the PLP process varied according to the filling density. After sintering, the magnets shown excellent shape retention, densities in the range of 6.9-7.4 g/cm3 and coercivities between 708-933 kA/m.

References:

[1] A.G. Popov, A.V. Shitov et al., The Physics of Metals and Metallography, 113, 331-340 (2012).

Novel synthesis and physical properties of $CoFe_2O_4$@$CoFe_2$ Core@Shell nanostructures

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In this work we present a study of $CoFe_2O_4$@$CoFe_2$ nanostructures of the core@shell type, which were synthesized by a new method without the use of special reagents or gases. First, we prepared small chitosan spheres with Co and Fe ions that were cross-linked ina solution with glutaraldehyde and NaOH. Second, the chitosan spheres were thermallytreated at high temperatures and in a vacuum, under a low pressure of 0.02 Torr, for 1h. The CO gas released from chitosan during the thermal treatment helped to reduce the $Fe^{3+}$and $Co^{2+}$to their zero valence state. Four samples were produced at temperaturesof 400, 500, 600 and 700 °C, and their structure, particle size, morphology and magneticproperties were studied. The X-ray difraction and Mössbauer spectroscopy showed thepresence of $CoFe_2O_4$and $CoFe_2$magnetic phases for samples prepared at 400 and 700 °C, respectively. For samples prepared at 500 and 600 °C we have obtained core@shell particles with crystallite sizes of 6.57-7.40 nm and 22.4-26.1 nm for $CoFe_2O_4$ and $CoFe_2$ phases, respectively. TEM images confrmed the presence of nanoparticles with core@shell confguration. Magnetic measurements showed that the $CoFe_2O_4$and $CoFe_2$phases, forthe sample treated at 600 °C, are coupled via exchange coupling, leading to a $(BH)_{max}$=0.67 MGOe. The estimate of the experimental thickness of the $CoFe_2$phase is 9.0 nm and is smaller than the critical size of 10.2 nm obtained by the Kneller-Hawig theory [1].

Acknowledgements:

M. S. Nunes thanks Capes for his scholarship. M. A. Morales thanks CNPq for his fellowship.

References:

[1] Kneller, E. F., Hawig, R. The exchange-spring magnet: a new material principle for permanent magnets. IEEE Transactions on Magnetics, 27(4), 3588-3560, 1991.

Positive unidirectional anisotropy in YIG/IrMn bilayers

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The phenomenon of exchange bias (EB), also called exchange anisotropy, or unidirectional anisotropy, results from the interfacial magnetic interactions at the atomic contacts between ferromagnetic (FM) and antiferromagnetic (AF) materials. Discovered over six decades ago [1], this phenomenon has been widely investigated theoretically and experimentally by means of several techniques and has also found applications in magnetoelectronic and sensor devices [2-3]. In FM/AF thin film bilayers the most known manifestation of the EB is the shift of the hysteresis loop along the field axis by some value Heb that varies inversely with the thickness of the FM layer. Usually, the exchange bias direction is opposite to the direction of the magnetic field applied during the sample growth, and is called negative EB. The reverse situation in which the hysteresis loop shifts in the direction of the field applied in the sample growth, called positive EB, was first observed in bilayers of Fe/FeF2 and is usually associated with an antiferromagnetic interfacial coupling at the FM/AF interface. We report an experimental study of the unidirectional anisotropy in bilayers made of the important ferrimagnetic insulator yttrium iron garnet (YIG) and the room temperature antiferromagnet IrMn. Measurements of the magnetization hysteresis loop in a wide temperature range and ferromagnetic resonance at room temperature revealed an unconventional positive exchange bias (EB). The presence of the positive EB is attributed to an antiferromagnetic coupling between the spins of the two layers at the interface.

[1] W. H. Meiklejohn and C. P. Bean, New magnetic anisotropy, Phys. Rev. 102, 1413 (1956).
[2] B. Dieny, V. S. Speriosu, S. S. P. Parkin, B. A. Gurney, D. R. Wilhoit, and D. Mauri, Giant magnetoresistance in soft ferromagnetic multilayers, Phys. Rev. B 43, 1297 (1991).
[3] J. Nogués and I. K. Schuller, Exchange Bias, J. Mag. Mag. Mat. 192, 203 (1999).

Reducing the Use of Costly Heavy Rare-Earth Elementsin Nd-Pr-Fe-B Magnets

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The demand for magnets for new high-frequency applications that must withstand high temperatures is growing, especially in motors for electric vehicles. Conventionally, this has been circumvented by partially substituting Nd for Heavy Rare-Earth Elements (HREE) such as Dy or Tb. This however increases raw-materials cost to the magnets production, since HREE are more scarce than Nd or Pr. Nevertheless, a new approach proposed by Hirota et al. [1] has shown that this element substitution does not necessarily have to be accomplished uniformly throughout the microstructure. Moreover if the additive (Dy) is cleverly located at the grain boundaries, an analogous effect is achieved to that when Dy is homogeneously distributed. One interesting method to accomplish that is by the so-called Grain Boundary Diffusion (GBD) which consists of a method of incorporating Dy through the grain boundaries of previously sintered magnets, under high temperatures, keeping the core of most grains intact. The main challenge of this technique is to obtain a shell-structure microstructure where the generation of high coercivity is related to a Dy-rich area on the grain periphery of the main phase ($Nd_2Fe_{14}B$), and a Nd-rich phase (Nd-Rich) on the grain boundaries [2]. In this work we report the feasibility of this method by using a Dy-rich eutectic alloy, which was previously embrittled to facilitate its comminution. The powdered alloy was then added to the surface of green compacts, or blended in the base alloy powder, in order to promote Dy diffusion through the grain boundaries simultaneously to the sintering process. The results show that the magnets in which Dy was added by the GBD method were able to achieve similar properties in the final magnet with 50% less addition of the HREE.

References:

[1] K. Hirota, H. Nakamura et al., IEEE Transactions on Magnetics, 42, 2909-2911 (2006).
[2] U.M.R. Seelam, T. Ohkubo et al., Journal of Alloys and Compounds, 617, 884-892 (2014).

Spin and charge DFT simulation on $CaFeO_3$ modified by oxygen vacancy

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The presence of oxygen vacancies in semiconductor oxides is not a novelty. Oxygen vacancies are n-type semiconductors, the Fermi''s energy modulates optical and electrical properties. For another hand, the Electron Paramagnetic Resonance (EPR) reported oxygen vacancy and magnetism for $CaTiO_3$material because of unpaired electrons promoting a charge transfer and photoluminescence effect [1].

Spin moments analysis in $CaFeO_3$indicates a residual up spin moment on the O atoms connecting the Fe atoms with 3.755 mB each. After oxygen vacancy, changes to the AFM order and 4.221 mB and -4.221 mB, respectively. Mössbauer technique identified magnetic changes on iron sites for $CaFeO_3$and $Ca_2Fe_2O_5$materials corroborating with model [2]. The introduction of one electron induces a FeM magnetic state. In contrast, O atoms have anisotropic spin moments distribution intensifying the magnetism on the Fe atom. The

Structural and magnetic properties of $CoFe_2O_4$ nanostructures produced by pulsed laser deposition

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In this work, we studied the magnetic and structural properties of cobalt ferrite nanostructures produced by pulsed laser deposition. We focus our investigation on spatial arrangements of nanoparticles with different degrees of porosity. The nanostructures were studied under different pressures in the deposition chamber: vacuum (10^{-6}Torr) and high pressures (0.1, 0.25, 0.5, and 1.0 Torr) of O_2. Scanning electron microscopy showed the growth of a thin film for deposition under vacuum and an arrangement of nanoparticles for the other pressures. It was observed that the distance and size of the nanoparticle clusters decrease with increasing deposition pressure, and after applying a pressure of 1.0 Torr, we observed a very porous system of nanoparticles. The high-resolution transmission electron microscopy analysis showed that the average size of the deposited nanoparticles is independent of the value of the deposition pressure. The magnetic results showed that the magnetic properties of nanoparticles are strongly influenced by pressure. The effects of magnetic interaction between nanoparticles generate, from a spin glass-like behavior for low deposition pressure to a behavior close to the expected for superparamagnetic systems for nanoparticles deposited under high pressure. Despite the pressure change in the deposition of the different cobalt ferrite samples, the electron diffraction results from the selected area provided the same crystallographic structure. The structural and morphological characterizations of the nanostructures deposited by PLD carried out in this work reveal important and unprecedented details of the nanostructures obtained using the PLD technique. In addition, we carried out a study of the capacitance of the cobalt ferrite nanostructures produced to check the potential of these nanostructures to be used in applications as supercapacitors.

Structural, magnetic and superconducting properties of NbC_x Nanoparticles produced by laser ablation in liquid

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Niobium carbide (NbC_x) is a highly promising material for superconducting applications due to its critical temperature ($T_c$of 8-12 K) slightly above that of Nb and high critical magnetic field (H_{c2}that can be bigger than 4 T).[1] Reducing the of this material leads to significant changes in the superconducting behavior, such as enhancing the critical field and/or the superconducting temperature.[2] Hence, In this study, NbC_xnanoparticles were produced using laser ablation in acetone, with varying lens-target distances and centrifugation velocities to control the particle . X-ray diffraction analyses revealed the presence of cubic NbC_xphase in all samples. Electron microscopy images showed spherical particles ranging in from few nanometers to hundreds of nanometers for all samples. Room temperature magnetization curves exhibited magnetic loops for all samples with saturation magnetization around tens of milli emu/g. At 2K, in addition to the magnetic component, a typical superconducting loop was observed for larger particles. A $T_C$of around 10 K was observed for all sample however, the diamagnetism below $T_C$decreased with particle , and the magnetic phase tended to dominate. The coexistence of superconductivity and magnetism in nanoparticles has garnered significant research interest in recent years, and we will discuss it in this work for NbC_xnanoparticles. Nanoparticles possess unique properties that differ from their bulk counterparts due to their large surface/volume ratio, and the interplay between superconductivity and magnetism in these systems can lead to find new matter properties and improve our understanding of the superconducting phenomenon.

Acknowledgements:
São Paulo Research Foundation (FAPESP) through Grant No. 2019/13678-1.References:

[1]G. F. Zou, et al., Nat. Commun., 2011, 2, 428.
[2] K. Bhattacharjee, et. Al., Phys. Chem. Chem. Phys., 2016, 18, 15218-15222.

Study of interparticle magnetic coupling by DPC-STEMof self-and magnetic-induced assemblies of Fe_3O_4 nanocrystals.

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Fe_3O_4nanocrystals (NC) can be used as patterns for fundamental studies of magnetic properties on the nanoscale because of two main characteristics: i) Fe_3O_4presents magnetocrystalline anisotropy being its direction of easy magnetization, thus cubic and spherical NC must have different magnetic behavior ii) the critical diameter of $Fe_3O_4$is around 25 nm, below this the NC is a magnetic monodomain. DPC-STEM could be a powerful tool to investigate magnetic phenomena because it allows obtaining information about interactions between electrons beam and different local magnetic orientations.[1] However, it should be noted that DPC is not trivial, because the TEM lenses are electromagnets and some TEM components are ferromagnetic material, thus is necessary to ensure that the magnetic field (MF) arising from these components does not affect the analyses.[2] Here, we aim to investigate how an external MF affects the formation of self-assembly of Fe_3O_4NC, taking into account the NC shape as well as to evaluate interparticle magnetic coupling at room and low temperatures (~196 °C) by DPC-STEM. Both spherical and cubic monodisperse 10 nm NC were obtained, and initial results of DPC-STEM of self-assembly NC on the TEM grid. STEM conditions are being improved. The next steps are to obtain the assembly of NC in thin films with and without the external MF.[3] FC-ZFC measurements also will be performed for corroborates DPC-STEM data and help in phenomenological interpretation.

Acknowledgments:
FAPESP (2021/03321-9) and FINEP (MQSMEML)

References :

[1] Naoya Shibata, et al.. Acc. Chem. Res. Vol 50, pp.1502-1512, 2017.
[2] Damien McGrouther, et al.. JEOLnews, Vol 49, 2014.
[3] Gurvinder Singh, et al.. SCIENCE. Vol 345, pp. 1149-1153. 2014.

Study of the Magnetocaloric effect on frustrated triangular trimers

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We study the thermodynamic features of frustrated magnetic trimers that are relevant to evaluate the capacity of magnetic refrigerarion. Exact expressions for the entropy change and magnetocaloric refrigeration rate are obtained considering both Ising and Heisemberg antiferromagnetic couplings between 1/2-spins. We show that magnetic frustration leads to an improved magnetocaloric refrigeration rate at low magnetic fields and temperatures. Further, a second region of enhanced refrigeration rate appears near the critical field separating frustrated and saturated groundstates. Both direct and inverse magnetocaloric effect are observed in the vicinity of his critical field. We also discuss on the possible role played by quantum fluctuations acting together with magnetic frustation to enhance the magnetic refrigeration capacity.

Superconductivity and charge density waves in transition metal chalcogenides NbS_3 and S-doped $ZrTe_2$

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Transition metal chalcogenides offer a wealthy playground to study the quantum effects related to low-dimensional systems. This family of Van der-Walls materials includes both the dichalcogenides (TMD) $MX_2$and trichalcogenides MX_3(TMT) variants (M = transition metal and X = Se, S, or Te). While the TMDs are known to feature quasi-2D properties, the TMTs materials are suggested to possess quasi-1D quantum properties, with the potential emergence of strongly correlated phenomena and topological effects, otherwise unattainable in other systems [1]. Despite being slightly distinct, the structural features of both TMD and TMT lead to charge density wave (CDW) ordering and highly anisotropic physical properties; in some cases, there is also coexistence between superconductivity and CDW in the TMD and TMTs [2]. In order to probe this coexistence, we have grown single crystals of intercalated TMT $NbS_3$and TMD S-doped $ZrTe_{2-x}S_x$using the isothermal chemical vapour transport, a technique recently developed by our group [3]. The physical and structural characterizations conducted in our crystals include X-ray diffraction, energy-dispersive spectroscopy, temperature and magnetic field dependent electrical resistance $\rho(T,H)$ and magnetization $M(T,H)$. Our results will be discussed focusing on the emergenceof superconductivity due to doping, intercalation, or off-stoichometry mechanisms, which may also lead to the suppresion of the CDW ordering.

Acknowledgements:

This project is financed by FAPESP (grant nos. 2022/02691-0, 2018/08819, 2021/14322-6, and 2021/08966-8), CAPES (88887.595960/2020-00) and CNPq.

References:

[1] A. A. Balandin, F. Kargar, T. T. Salguero, and R. K. Lake, Mater. Today 55, 74 (2022).

[2] X. Zhu, H. Lei, and C. Petrovic, Phys. Rev. Lett. 106, 246404 (2011).

[3] L. E. Correa, L. R. de Faria, R. S. Cardoso, N. Chaia, M. S. da Luz, M. S. Torikachvili, and A. J. S. Machado, J. Cryst. Growth 595, 126819 (2022).

Superconductivity in Te-deficient ZrTe2

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We present measurements of structural, electrical, and thermoelectric potential on high-quality single crystals of ZrTe1.8 grown from isothermal chemical vapor transport. Our results indicate that despite being Te-deficient and sharing the same structure as the non-superconducting ZrTe2, ZrTe1.8 exhibits superconductivity below 3.2 K. The temperature dependence of the upper critical field (Hc2) deviates from the behavior expected in conventional single-band superconductors and is best described by an electron-phonon two-gap superconducting model with strong intraband coupling. Seebeck potential measurements of the ZrTe1.8 crystals suggest that the charge carriers are predominantly negative, consistent with ab initio calculations. Moreover, our DFT-based first-principles calculations reveal that the inclusion of Te vacancies in ZrTe2 unexpectedly leads to the formation of localized Zr-d bands and a density of states peaks at the Fermi level, possibly promoting electronic instabilities at the Fermi level and increasing the critical temperature according to the standard BCS theory. These findings suggest that Te deficiency can fine-tune the electronic structure to support superconductivity in TMDs and highlight the role of defects in promoting the stability of the superconducting ground state.

Superconductivity in Terbium intercalated $ZrTe_2$

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$ZrTe_2$ is a part of the transition metal dichalcogenide family, comprised of two-dimensional materials where van der Waals forces create a layered structure in which foreign species may be intercalated between chalcogenide layers. $ZrTe_2$ in particular, may be regarded as a Dirac semimetal where multi-gap superconductivity has been previously reported for Cu (T_c= 9.0 K), Cr (T_c= 5.4 K) and Ni (T_c= 4.0 K) intercalated specimens, as well as for Tellurium deficient samples (Tc = 3.2 K). Due to the possible coexistence between superconductivity and non-trivial topology, $ZrTe_2$ is a good candidate for probing of the interplay between different emerging quantum states, and the intercalation of atoms with incomplete *f* electron shells may be a good path to unveil the intricacies of this system and gain insight on the type of pairing mechanisms that induce superconductivity in this compound. In this work we prepared single crystals of Terbium intercalated $ZrTe_2$ single crystals, and we show superconductivity is developed with T_c in the vicinities of 5.0 K. The single crystals are characterized by means of X-ray diffractometry and Energy Dispersive Spectroscopy, and its superconducting properties are characterized by means of DC electrical resistivity and magnetization measurements.

Acknowledgements

This project was financed by FAPESP under projects 2018/08819, 2021/14322-6, 2021/08966-8 and 2022/12392-0, CAPES under project 88887.595960/2020-00 and CNPq.

References

1 – Phys. Rev. B. **95** (2017) 144505.
2 – J. Alloys Compd. **907** (2022) 164477.
3 – J. Phys. Chem. C. **127** (2023) 5162-5168.

Synthesis and Characterization of Magnetic FluidBased on Copaifera Officialis L. Oil

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This study reports the successful preparation, characterization with temporal analysis of an organic magnetic fluid using superparamagnetic nanoparticles coated in essential oil of Copaifera Officialis L.. Therefore, the Fe_3O_4nanoparticles will be synthed by the coprecipitation method by hydrolysis in an alkaline solution. Ferrofluid will be prepared by dispersing magnetic nanoparticles, at five different concentrations, in the in natura oil. For the samples characterization measurements were taken of: UV-vis, FTIR, NIR spectrophotometry, XRD and Magnetic Susceptibility, the temporal analysis was recorded periodically, observing the possible stability of the samples. The Molar Susceptibility and XRD data show that SPIONs have an average diameter of 21 nm and are highly susceptibleto magnetic fields. The analysis by spectrophotometry in UV-Vis and NIR indicate that the magnetic fluid has the properties associated with Amazon natural oil. the samples showed good stability, the data indicate that the concentrations of 13% and 16% are the most suitable for the synthesis of magnetic fluids based on the vegetable oil of Copaifera Officialis L. [1,2].

Acknowledgments

This was financially and academic support offered by Postgraduate Program in Nanoscience and Nanobiotechnology from University of Brasilia -BR and Federal Institute of Education, Science and Technology of Rondônia -Brazil

References:

[1] V.F. Veiga Jr and A. C. Pinto, “. O GÊNERO Copaifera L.”, Química Nova, Rio de Janeiro, 2002, v. 5, n. 2, p. 273-286.

[2] Laurent, D. Forge, M. Port, A. Roch, C. Robic, L. Vander Elst, R. N. Muller, “Magnetic iron oxide nanoparticles: synthesis, stabilization, vectorization, physicochemical characterizations, and biological applications”, Chemical reviews, 2008, v. 108, n. 6, p. 2064-110.

Synthesis and characterization of the electricalproperties of LaNiO3 nanostructures by electrospinning.

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Rare earth-nickelates are a class of electronically correlated oxides, where their physical properties are directly related to their crystalline structure. These compounds crystallize in the rhombohedral or orthorhombic distorted perovskite structure, where structural distortion or chemical substitution directly influences their physical properties [1]. LaNiO3 crystallizes in rhombohedral-distorted perovskite structure and has a metallic behavior from2 to 300K. The characteristics of high electrical conductivity and high thermal stability observed experimentally for LaNiO3 nanoparticles have motivated the study of its field emission properties. In particular, the use of conductive nanotubes or nanowires can also function as localized sources of electrons. In this context, this work produced LaNiO3 nanowires by electrospinning to evaluate their transport and field emission properties. For this, the wires were produced from a La-Ni precursor solution, with a viscosity of ~1000cP. Electrospinning was performed and they are being characterized by X-ray diffraction, scanning electron microscopy (SEM) and electrical transport measurements. Preliminary characterizations revealed that these compounds crystallized in the rhombohedral-distorted perovskite (R-3C) LaNiO3 phase, without the presence of additional phases. SEM images showed that the samples are shaped like nanowires with lengths of several micrometers (5 to 7μm) and an average diameter of around 150nm. Transport properties are currently being evaluated.

Acknowledgements:

The authors are grateful to the Multiuser Central Facilities (UFABC) for the experimental support, CAPES for funding the scholarship and FAPESP (2022/11278-9).

References:

[1] S. Catalano, et. al. Reports Prog. Phys., 81, 4, 046501 (2018).
[2] J. H. H. Rossato et al., ACS Appl. Nano Mater., 5, 3, 3394-3405, (2022).

Synthesis, characterization and application of poly(allylthiourea-acrylamide) with restricted access in Lead Preconcentration with Simultaneous Exclusion of Macromolecules in foods using FAAS

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The use of magnetic nanomaterials for ion preconcentration has received significant attention in recent years due to the absence of filtration and/or centrifugation steps in extraction procedures. However, due to the high aggregation power and lack of selectivity of these magnetic cores, the modification of their surface with ionically imprinted magnetic polymers with restricted access (IIPs-RAM) has been highlighted for this purpose[1]. Thus, an IIP-HEMA-RAM was synthed using acrylamide and allylthiourea as functional monomers in the presence of BSA and HEMA as hydrophilic co-monomers. To evaluate the effect of chemical imprinting and exclusion of macromolecules, control materials, such as: unprinted and without restricted access, were also synthed. All materials were characterized by ATR-FTIR, SEM, MET, BET, DRX and TG to prove the formation of polymers and their structural and morphological characteristics. Optimizing factors that affect MSPE, such as pH, preconcentration and elution time, adsorbent mass, and sample volume, will be optimized univariately. From the ATR-FTIR spectra, it was possible to prove the magnetism of the material due to the presence of the band at 540 cm^{-1}, as well as the formation of a core-shell attributed to the band at 1100 cm^{-1}, characteristic of the Si-O bond and the band at 1641 cm^{-1}attributed to vinylic groups in the polymeric matrix. From the pH and zero charge point studies carried out for the IIP-HEMA-BSA material, more significant adsorption was observed at pH 6.0, proving that adsorption of the Pb2+ ion in the material occurs through interaction with acrylamide and allylthiourea monomers. Studies of selectivity, interferents, adsorption kinetics, and isotherms, as well as the application of the method in food samples, will be carried out.

Acknowledgments: CAPES, CNPq, and UEL.

References:[1] Suquila, F. A. C. e Tarley, C. R. T., Talanta, 202, 460-468, (2019).

SYNTHESIS OF BARIUM HEXAFERRITE PARTICLES PREPARED BY SOL-GEL COMBUSTION METHOD

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Magnetic materials have always attracted human attention for being convenient in several applications of electronic devices such as permanent magnets and recently in treatments for diseases such as cancer and also for the release of drugs that can be conducted and limited in a given location by means of magnetic field. In this context, this work consists of the synthesis of barium hexaferrite particles ($BaFe_{12}O_{19}$) that were obtained through the sol-gel combustion method [1], employing as precursors barium nitrate and hydrated iron nitrate nona, besides that, citric acid and ammonium hydroxide were added to the solution. Phase formation was investigated by thermogravimetric analysis (TGA), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results reveal that the formation of the single-phase powder occurs with increasing temperature.

Acknowledgements:

The Fundação de Apoio à Pesquisa do Distrito Federal (FAPDF) and the University of Brasilia (UnB) for the incentive to research.

References:

[1] A. Mali and A. Ataie, "Influence of the metal nitrates to citric acid molar ratio on the combustion process and phase constitution of barium hexaferrite particles prepared by sol-gel combustion method," Ceramics International vol. 30, pages 1979-1983 (2004).

Theoretical DFT/HSE06 study of crystalline and electronic structure of X0.125Mg0.875B2 materials (X = Nb, Ni, Fe)

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MgB2 material is widely applied in superconductors studies and generation of electronic devices, . The modification of these material is reported in literature with transitions metals in Mg site. Fabrication of superconductors wires of low-cost with MgB2 materials is reported as a promissory option to application in electronic devices and in magnetic resonance devices. With hexagonal P6/mmm space group, these material presents a versatile structure to modifications with transitions metals in Mg site [1].

The computational simulations in work were performed on DFT/HSE06 level in software CRYSTAL17, and VESTA viewer. The Mg, B, Ni, Fe and Nb atoms were described by 8-511G, m-6-311G(d), TZVP, s8411p6411d441 and 986-31(631d)G Gaussians basis sets respectively. The Mg0.875X0.125B2 (X=Nb, Fe and Ni) material was simulated in a 2x2x2 supercell in Mg site. The electronic profile based on the Electronic Band Structure (EBS) and Density of States (DOS).

Quantum simulations of MgB2 material compared with cell parameters a = b = 3.073 Å andc= 3.553 Å reported in literature [2], presents a good crystalline structure. Ni, Fe and Nbdoping presents low distortions in a and b parameter. The considerable distortion is the effect of Nb, Ni and Fe doping is the contraction in parameter c, denoting an approximationof layers. In EBS analysis is observed the effect of magnetic cation in structure in Fe and Nidoped material is that the paired electrons in beta channel forms the Dirac Cone in symmetry point A, while in Nb the Dirac Cone is observed in alpha and beta channel in Γand A point. In Fe and Ni doped materials, the profile of Dirac Cone is more defined and theintersection of Bands is most clear.

Acknowledgements:

Acknowledgements to CAPES and UEPG by support.

References:

[1] HUŠEK, I. et al. Ceramics International, vol.49, n.7, p. 11178-11183, 2023.

[2] ZHANG, Yu XU, Xiaojie. Physica C: Superconductivity and its Applications, vol. 573, p. 1353633, 2020.

Theoretical investigation on magnetic and magnetocaloric effect of $Gd_{0.2}Tb_{0.2}Dy_{0.2}Ho_{0.2}Er_{0.2}Al_2$ high entropy alloy

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High entropy alloys are a group of advanced materials composed of five or more metallic elements mixed in roughly equal proportions. The concept behind HEAs is that by combining multiple elements in equal proportions, it is possible to create materials with high entropy or disorder, which can lead to improved mechanical, thermal, and electrical properties [1].

In this work, we report theoretical calculations of thermal and magnetic quantities such as magnetization and heat capacity, as well as magnetocaloric potentials (isothermal magnetic entropy (ΔS_T) and adiabatic temperature (ΔT_{ad}) changes) in the ferromagnetic high entropy alloy $Gd_{0.2}Tb_{0.2}Dy_{0.2}Ho_{0.2}Er_{0.2}Al_2$using a microscopic model. The model hamiltonian includes contributions from the Zeeman effect, crystalline electrical field anisotropy, and exchange interactions among the five different rare earth sublattices (Gd-Gd, Gd-Tb, Gd-Dy, etc.). Most of these were found by considering previous studies on pseudobinary compounds such as $Gd_{1-x}Dy_xAl_2$[1], $Gd_{1-x}Tb_xAl_2$[2], $Er_xTb_{1-x}Al_2$[3], $Ho_{1-x}Dy_xAl_2$[4], etc.

Experimental data [5] show that the Curie temperature is approximately the average of the individual components RAl2 (R = Gd, Tb, Dy, Ho, and Er). The theoretical results are in agreement with the available experimental data [5].

References:
[1] J. Y. Law and V. Franco, Journal of Materials Research, vol. 38, issue 1, 2023. DOI: 10.1557/s43578-022-00712-0
[2] B. P. Alho et al., International Journal of Refrigeration, vol. 37, p. 297-302, 2014. DOI: 10.1016/j.ijrefrig.2013.07.012
[3] P. O. Ribeiro et al., Journal of Alloys and Compounds, vol. 563, p. 242-248, 2013. DOI: 10.1016/j.jallcom.2013.02.068
[4] Y. Mudryk et al., Metals, vol. 10, p. 1662, 2020. DOI: 10.3390/met10121662
[5] P. O. Ribeiro et al., Journal of Magnetism and Magnetic Materials, vol. 544, p. 168705, 2022. DOI: 10.1016/j.jmmm.2021.168705
[6] P. K. Jesla et al., IEEE Transactions on Magnetics, vol. 58, no. 2, 2022. DOI: 10.1109/TMAG.2021.3088470

Theoretical study of magnetism and cooling capacity of amorphous alloysformed by the series $Gd_{54}Fe_{36}B_{10-x}Si_x$.

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The magnetocaloric effect (MCE) - variation of the temperature of a magnetic material under the variation of the external magnetic field can be characterized by the variation of the cooling capacity (RC) and the magnetic entropy. We observe in general that the RC of an amorphous alloy is greater than its equivalent in the crystalline phase. Furthermore, structural disorder minimizes hysteresis losses, but the existence of local random anisotropy due to structural disorder can lead to more complex magnetic structures. Some consequences, for example, are the reduction of spontaneous magnetization and the reduction of the magnetic ordering temperature. In this work, we will investigate the magnetic and magnetocaloric properties of the amorphous alloys formed by the Gd54Fe36B10-xSix series. From an experimental point of view, these systems have been reported to have high values for refrigerant capacity and interesting magnetocaloric properties associated with ferrimagnetic ordering. We propose a theoretical model described by two coupled magnetic sub-lattices of localized spins in the presence of an applied magnetic field based on the Handrich approximation to study these systems. By means of this model, we calculate the magnetization and the isothermal entropy change as functions of the temperature under the change of the applied magnetic field. We will try to understand the role of the replacement of B by Si and the influence on magnetic entropy and the magnetocaloric effect of these systems. Furthermore, the study is important to understand the physical mechanisms involved in the ferrimagnetic transitions of amorphous alloys.

Thermoelectric response of Co-rich flexible ribbons

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$Fe_3Co_{67}Cr_3Si_{15}B_{12}$ribbons with a high degree of flexibility and excellent corrosion stability were produced by the rapid quenching technique. Their structural, magnetic, and thermomagnetic (Anomalous Nernst Effect) properties were studied both in an as-quenched (NR) state and after stress annealing during 1 h at the temperature of 350 ◦C and a specific load of 230 MPa (AR). X-ray diffraction was used to verify the structural characteristics of our ribbons. Static magnetic properties were explored by inductive technique and vibrating sample magnetometry. The thermomagnetic curves investigated through the Anomalous Nernst Effect are consistent with the obtained magnetization results, presenting a linear response in the thermomagnetic signal, an interesting feature for sensor applications. Additionally, Anomalous Nernst Effect coefficient S_{ANE}values of 2.66 μV/K and 1.93 μV/K were estimated for the as-quenched and annealed ribbons, respectively. The interplay of the low magnetostrictive properties, soft magnetic behavior, linearity of the thermomagnetic response, and flexibility of these ribbons place them as promising systems to probe curved surfaces and propose multifunctional devices, including magnetic field-specialized sensors.

Acknowledgements:

CNPq, CAPES e INCT-SpinNanoMag.

References:

CORREA, MARCIO A.; FERREIRA, ARMANDO ; SOUZA, ARTHUR L. R. ; DANTAS NETO, JOÃO. M. ; BOHN, FELIPE ; VAZ, FILIPE ; KURLYANDSKAYA, GALINA V. . Anomalous Nernst Effect in Flexible Co-Based Amorphous Ribbons. SENSORS, v. 23, p. 1420, 2023.

Thermomagnetic study of Co_2FeAl Heusler alloy basedon graphene-doped flexible ceramics.

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GnP-doped Al_2O_3flexible tapes were produced through Tape Casting technique and used as substrates to produce Co_2FeAl/GnP-doped Al_2O_3heterostructures. The Co_2FeAl layers were grown using Magnetron Sputtering with a thickness of 30 nm. The integration of Tape Casting and Magnetron sputtering techniques allowed us to explore the thermomagnetic properties of the heterostructure through the Anomalous Nernst Effect. The structural and morphological properties of the substrates and the magnetization curves support the thermomagnetic results. The CoFeAl ferromagnetic films presented isotropic magnetic behavior, which was reflected in the thermomagnetic curves through the stability of the curve’s shape irrespective of the thermal gradient and angle of the external magnetic field used during the measurements. Our findings open new possibilities to produce flexible heterostructures with multifunctional properties, where the versatilities of the Tape casting and magnetron sputtering techniques bring new routes to build nanostructured materials.

The role of terminal ligands on the slow relaxation of magnetisation and luminescence thermometry of dinuclear Nd^{III} complexes

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Single-Molecule Magnets (SMMs) are at the forefront of the next generation of molecular-based electronics.[1] As their state-of-the-art is mainly based on heavy lanthanide ions, examples of light lanthanide-based complexes e.g., Nd^{III}, fall far behind due to the strong contribution of quantum-tunnelling of magnetisation (QTM).[2] Besides the slow relaxation of magnetisation, the luminescence of multifunctional SMMs can be used as a tool for ***in situ***temperature measurements through luminescence thermometry.[3] To help understand how the magnetic and luminescent features of Nd^{III}-based SMMs can be enhanced by structural modifications, herein, two dinuclear complexes, $[Nd_2(valdien)_2(X)_2]$ (X = $acac^-$ {1-Nd_2} or NO_3^- {2-Nd2}) (H_2valdien = N^1,N^3-bis(3-methoxysalicylidene)diethylenetriamine, $acac^-$= acetylacetonate), are presented. The complexes display field-induced slow relaxation of magnetisation, but for {2-Nd_2}, QTM has a lower contribution because the longer Nd^{III}-$O_{terminal}$bond distances induce a weaker equatorial crystal field component. Taking advantage of the Nd^{III}near-infrared emission, the systems were used to develop temperature probes displaying maximum thermal sensitivity of 0.52% K^{-1}at 40 K {2-Nd_2}. Therefore, this approach can enable mapping strategies to boost the luminescent and magnetic performance of similar multifunctional SMMs.

Acknowledgements:The Canadian Foundation for Innovation, the Natural Sciences and Engineering Research Council of Canada, and Sa˜o Paulo Research Foundation (Grant No. 2021/09755-0, 2019/23763-6, 2021/06326-1, and 2021/08111-2).

References:

[1] R. Marin, G. Brunet, M. Murugesu, Angew. Chem. Int. Ed. 60, 1728 (2021).
[2] Y-C. Chen, X-S. Huang, J-L Liu, M-L. Tong, Inorg. Chem. 57, 11782 (2018).
[3] D. Errulat, R. Marin, D. A. Gálico, K. L. M. Harriman, A. Pialat, B. Gabidullin, F. Iikawa, O. D. D. Couto Jr., J. Moilanen, E. Hemmer, F. A. Sigoli, M. Murugesu, ACS Cent. Sci. 5, 1187 (2019).

Unraveling the spin current flow in Bi layers

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Although heavy metal materials, due to their large spin-orbit interaction, would be expected to act as an efficient converter of spin current to charge current, Bismuth does not obey this rule. Heterostructures of yttriumiron garnet (YIG)/Bi are investigated by means of the spin pumping technique and the results show that Bi exhibits negligible spin-charge conversion. In addition to not converting spin to charge, Bi exhibits an exotic spin relaxation mechanism, which is driven by its large diamagnetic response. By considering a nonlinear stochastic model of diffusing spin particles, the results show that the Bi layer acts as a binary classifier device that categorizes the pumped spins into relaxed spins and those that flow upwards and eventually reach the toplayer of Pt. This shows that the investigation reported here opens different perspectives involving fundamental aspects in the fields of machine learning and condensed matter physics.

UNVEILING THE POLAR PROPERTIES ON BARIUM BISMUTHATE PEROVSKITE THIN FILMS WITH DISTINCT Ba/Bi RATIO

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In this work, we investigate the domain structure of Ba(1-x)BixO3 (x=0.2, 0.5, 0.7) synthesized via chemical solution deposition (CSD) method. The samples were named as BBO37, BBO55 and BBO82 according to distinct Ba/Bi ratio. The electrical response of the as-prepared BBO thin films was explained based on its (micro)structure, using a combination of Transmission Electron Microscopy (TEM), and X-ray diffraction (XRD). The piezo-ferroelectric coupled behavior, observed for BBO, was demonstrated, and elucidated in terms of point defects (e.g., oxygen vacancies and cation defects). All the films crystallizein a rhombohedral BaBiO3 structure with a R-3R space group without any deleterious secondary phase. TEM micrograph images indicate that the Bi content significantly influence grain morphology. Additionally, the hysteretic strain, dielectric, ferroelectric, and piezoelectric properties of BaBiO3 thin films are strongly dependent on the electronic structure. Hence, the electrical response of BaBiO3-based ceramics is determined by the presence of point defects (e.g., oxygen and bismuth vacancies) as well as grain morphology, which can be controlled by stoichiometric changes (Ba/Bi ratio). Sample BBO55 showed a distinguished preferred orientation along the (341) plane with a low associated coercive field (63.55 kV/cm), free of imprint and with no polarizations gap compared to samples BBO37 and BBO82. Sample BBO55 presented the highest piezoelectric (d33-eff≈ 45.7 pm/V) and ferroelectric (Pr≈13.67 µC/cm2) responses at room temperature. This behavior can be ascribed to a highly textured film, which generates less strain throughout the crystal lattice facilitating the polarization switching mechanism and improving piezo/ferroelectricity.

YIG films produced by the metalorganic decomposition method (MOD) lithographed by focused ion beam (FIB)for the study of spintronic phenomena

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Yttrium iron garnet films - YIG (Y3Fe5012) have been widely used in the study of spintronic phenomena, induced by spin waves. Since silicon is highly compatible with microelectronic technologies, the continuous search for optimizing the process for obtaining these films has become a reality. This, thanks to the existing structural incompatibility between this substrate and the YIG. The manufacture of these films by chemical methods and, in particular, on silicon substrate, is still a great challenge. For this reason, large studies presented in the literature use conventional methods for the production of YIG films for these purposes. This work, in turn, aims to present quality YIG films in terms of morphology, as well as structural and magnetic properties, for studies of spintronic phenomena. The films were produced from the metalorganic decomposition method (MOD), being deposited by spin coating. The films were annealed at 850°C and characterized by XRD, SEM, Raman and FMR. The films showed a single-phase YIG structure, with a line width of 93 Oe in the ferromagnetic resonance (FMR) spectrum. Arrangements of hollow and non-hollow nanopillars were lithographed using the focused ion beam - FIB, becoming a perspective for spintronics studies.

Z-Magnetism, spintronics, superconductivity and complex magnetic nanostructures

Activation of alkane C-H bonds by Mn porphyrin-based catalysts immobilized onto SiO_2, SBA-15, and HMS materials as cytochrome P450 models

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Cytochromes P450 are heme-containing monooxygenase enzymes that carry out the activation and selective oxygenation of inert C-H bonds of alkanes under mild biological conditions[1]. P450-like catalysts based on Mn-porphyrins (MnP) heterogenized onto silicas are actively sought after as biomimetic models[1,2]. In these systems the MnP mimics thereactivity of the heme group whereas the silica support models some of the protein roles. Hereon we describe the heterogenization of two structurally related MnPs onto ordinary chromatographic amorphous silica gel (SiO_2) and mesoporous silicas SBA-15 and HMS: Mn(III) *N*-methylpyridinium-2-ylporphyrin (MnP2) was electrostatically supported on non-modified silicas, whereas Mn(III) 2-*N*-pyridylporphyrin (MnP1) was covalently immobilized onto chloropropyl-functionalized silicas. All six materials were investigated as P450 models for cyclohexane (Cy-H) oxidation by iodosylbenzene. The SiO_2-Cl/MnP1 material was the most robust, efficient and selective (~60%) for Cy-H oxidation, with total yield of 89%. Recycling studies showed that the SBA-15- and HMS-based materials were somewhat more susceptible to oxidative degradation of MnP. Amorphous silica materials (SiO_2-Cl/MnP1 and SiO_2/MnP2) maintained a high overall yield, with some loss in efficiency on the 3rd cycle of use. Cyclohexanol selectivity was little affected by catalyst recycling. In this work, we show a comparison of the catalytic efficiency of the materials and the role of the silica support onto catalyst reactivity and P450 modeling.

Acknowledgments:
CNPq, CAPES, FINEP, Fundação Araucária, UFPR, UFPB, INCT-CiMol.

References:
[1] PINTO, V. H. A. *et al.*, Applied Catalysis A: General, 526, 9-20 (2016).
[2] PINTO, V. H. A. *et al.*, Dalton Transactions, 49, 16404-16418 (2020).

A new biopolymer obtained from glycerol and maleic anhydride using a polythiol as a crosslinking agent by photopolymerization

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Nowadays, glycerol and maleic anhydride emerge as proponents of green inputs for the production of biopolymeric materials [1]. In addition, the use of polyols for the production of biopolymers with properties similar to those from non-renewable sources has been producing materials of high technological interest [2]. Thus, in the present work, it was performed the synthesis of a pre-polymer using glycerol and maleic anhydride, which subsequently was mixed with the polythiol pentaerythritol tetrakis(3-mercapto propionate). The final polymer was obtained by photopolymerization (using the photoinitiator 2,2-dimethoxy-2-phenylacetophenone and UV light). The mid-infrared absorption spectroscopy (MIR) analysis suggested the formation of an ester (from pre-polymer) and the consumption of double bonds of the alkenes, suggesting the occurrence of the thiol-ene reaction. The final material presented luminescent properties, showing a great potential to use as sensors.
Acknowledgments: FAPESP (grants 2022/03489-0 and 2021/02152-9) and CNPq (grants 303247/2021-5 and 150233/2022-1).

References:

[1] R. T. Alarcon, B. B. C. Holanda, A. R. Oliveira, A. G. Magdalena, G. Bannach, Revista Virtual Chemistry, vol. 9, p. 163-175 (2016).

[2] T. O. Machado, C Sayer, P. HH. Araujo. European Polymer Journal, v. 86, p. 200-215, 2017.

Antistatic and antimicrobial packaging of PHBV/PLA blends-based graphene nanoplatelets and glassy carbon nanocomposites

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The generation and accumulation of electrostatic charges is one of the main problems encountered in the production and operation processes in the electronics industry [1]. Antistatic packaging is used to protect electronic devices against damage caused by electrostatic discharges and these packages are generally produced with non-biodegradable polymers of fossil resource [1]. Thus, the production of packaging obtained from biodegradable materials constitutes a promising research area. In this work, two biopolymers were chosen: poly (lactic acid) (PLA) and poly (3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), both of renewable origin. Glassy carbon (GC) and graphene nanoplatelets (GNP) were chosen as antistatic agents. PLA/PHBV blends (80/20) with the addition of 0.1 and 0.5 wt% of GC and 3 wt% of GNP were prepared in a twin screw extruder (AX Plásticos, model AX16:40DR). Specimens were prepared by injection molding (Battenfeld, model Plus 350/75). The nanocomposites were characterized by tensile test, impedance spectroscopy (Impedance/Gain-phase Analyzer, model Solartron SI 1260) and Minimum Inhibitory Concentration test (using the following microorganisms: Escherichia coli ATCC 25992 and Staphylococcus aureus ATCC 6538). The addition of GNP and GC caused an increase in the elastic modulus of the nanocomposites. All compositions showed adecrease of 9 orders of magnitude in electrical resistivity (104 Ω.m), presenting the electrical properties for application as antistatic packaging. The compositions showed MICvalues greater than 70%, demonstrating their antimicrobial potential. PLA/PHBV blend withthe addition of 0.1 wt% of GC and 3 wt% of GNP showed optimization of properties, withhigher elastic modulus, low electrical resistivity and antimicrobial activity.

Acknowledgments:

The authors thank FAPESP (Process 2020/12501-8) for financial support.

Reference:

[1] T. F. Silva, et al. Journal of Applied Polymer Science, 136, 47273 (2019).

Biogenic synthesis of silver nanoparticles usingbacteria for agricultural applications

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With the increase in pesticide resistance, silver nanoparticles (AgNPs) have been widely used, mainly in the agricultural sector. The biogenic synthesis of these nanoparticles has a high antimicrobial potential and stimulates plant growth and nutrition. In the present study the AgNPs were synthesized using ***Bacillus thuringiensis***, ***Bradyrhizobium japonicum*** and ***Azospirillum brasilense***, and 1 mM silver nitrate as metallic precursor. After the synthesis, chemical characterization was performed to determine the hydrodynamic diameter, polydispersion index (PDI) and zeta potential. The antimicrobial effect with microorganisms ***Escherichia coli***, ***Staphylococcus aureus***, ***Pseudomonas aeruginosa*** and ***Candida albicans*** were evaluated by disk diffusion and minimum inhibitory concentration (MIC). Thein vitro biological activity was evaluated against the phytopathogenic fungi ***Fusarium oxysporum***, ***Fusarium graminearum,*** and ***Sclerotinia sclerotiorum***. Cytotoxicity and genotoxicity were evaluated with the 3T3 cell line by MTT and comet assays. Effects on seedgermination and growth were investigated with broccoli seeds. AgNPs showed a mean hydrodynamic diameter of 79.70±1.10 nm, PDI 0.51±0.01 and zeta potential of −33.30±0.78mV. Disk diffusion and MIC assays showed that AgNPs inhibited pathogenic microorganisms. AgNPs showed inhibitory potential against phytopathogens with greatereffect on ***S. sclerotiorum***. The IC 50 was found at a concentration of 33%, with no indicationof genotoxicity. Regarding the effects on seed germination, no significant differences wereobserved. This study paves the way for further investigations on biogenic AgNPs.

Acknowledgements:

University of Sorocaba (UNISO) and State University of São Paulo (UNESP).

References:

[1] MUSTAPHA, T. et al. A Review on Plants and Microorganisms Mediated Synthesis of Silver Nanoparticles, Role of Plants Metabolites and Applications. International Journal of Environmental Research and Public Health, v. 19, n. 2, 2022.

CaO-Nanofluid from Mytella falcata shells applied to enhanced oil recovery

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The availability of petroleum is declining, and the current technology is capable of recovering between 20 and 40 % of oil stored in reservoirs [1]. The present work aimed to produce and test nanofluids for the enhanced oil recovery (EOR), targeting the increase in recovery from mature reservoirs. Nanofluids (NF) were produced using nanoparticles of calcium oxide from Mytella falcata shells, derived from irregular discard in Alagoas State, which were used at concentrations of 0.05, 0.1 and 0.5 % (m/m), through the twostep method, where, after the nanoparticle conception as a powder, they are dispersed in the base fluid, at the proper concentrations. The nanofluids produced were evaluated in terms of nanoparticles surface composition, contact angle, recovery efficacy in a flooding assay using a simulated porous medium and alteration of conductivity. The XRD results indicated a characteristic Ca-O peak at 2θ = 32.1º, and the FTIR showed a strong stretch at 712 cm-1, this region is characteristic of CaO absorption bands. The wettability alterations were observed via contact angle analysis between the NF droplet and the saturated rock sample. The nanofluid at a concentration of 0.05 % had a decrease of 41.95º; 0.1 % reduce the angleby 36.57º; and at 0.5 %, there was a 44.12º decrease related to the base fluid, distilled water. This effect indicates a shift of wettability from oil-wet to water-wet, favouring the recovery of the oil impregnated into the rock. There was also

a rise in the base fluid conductivity, increasing the Brownian movements of the nanoparticles in the system. At last, the efficiency of oil recovery was improved by 8.5%, demonstrating the potential of the method in EOR.

Acknowledgements:

All authors thank CAPES for the financial support.

References:

[1]LASHARI, N.; GANAT, T. Emerging applications of nanomaterials in chemical enhanced oil recovery: Progress and Perspective. Chinese Journal of Chenical Engineering, p. 1995-2009, 2020

CATIONIC EXCHANGE IN TITANATE NANOTUBES AND EVALUATION OF PHOTOCATALYTIC POTENTIAL IN DEGRADATION OF METHYLENE BLUE DYE

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Water pollution with dyes has become a very frequent problem, bringing severe consequences to natural resources and human health, being often carcinogenic and very resistant in natural conditions and the treatment of polluted waters is a major challenge since these products are not biodegradable. Among the most appropriate techniques for dye degradation is photocatalysis. In this work was performed the synthesis of Titanate nanotubes by the microwave-assisted alkaline hydrothermal method and the cation exchange of Na^+ions by the metallic cations $Sr^{2+}$and Co^{3+}, the photocatalytic activity of the catalysts synthed in the degradation of the methylene blue dye.

TiNTs were synthed using microwave-assisted alkaline hydrothermal synthesis, using TiO_2as precursor and NaOH at 10 mol.L^{-1}. Ion exchange was performed by suspending NaTiNTs, previously synthed, to solutions of cobalt and strontium nitrates. Photolysis and photocatalysis were performed in a closed box with LED lamp (30 W), in which 50 mg of the catalyst was added to a 0.1 mmol solution. L $^{-1}$of the dye. Throughout the procedure the solution was oxygenated and kept under agitation at room temperature. Samples were collected during the process at specific times between 0 to 120 minutes.

The techniques used for the structural analysis of the materials were Raman and FT-IR. The results of the photolysis and photocatalysis tests were analyzed by UV-vis spectroscopy and the kinetic study with the degradation rate was performed. The synthesis of Titanate nanotubes from the hydrothermal method was efficient, as well as the ion exchange process of Na^+ions by $Co^{3+}$and Sr^{2+}ions. The Raman spectrum found that the structure of the nanotubes remains intact after their insertion. The pure and exchanged TiNTs showed good photocatalytic activity in the degradation of methylene blue.

Characterization of malachite pigment interaction with linseed oil hydrolysis/oxidation products in paints under accelerated aging - reflections on the conservation of cultural heritage

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The interaction between inorganic pigments and hydrolysis/oxidation products of polymerizable oils can be harmful to cultural heritage paintings [1]. In this work, the interaction of the pigment malachite green ($Cu_2(OH)_2CO_3$) with linseed oil submitted to accelerated aging was investigated [2]. This preparate ink was applied onto a 10x3 cm linen and aged at 105 °C for 5, 24, 168, 360 and 720 h corresponding to 2.25; 10.8; 75.7; 162 and 324 years of aging at 25 °C, respectively. For the sample aged 24 h, FTIR studies revealed new bands at 1716 and 1700cm^{-1}, indicating the presence of carboxylate groups, which is related to the hydrolysis/oxidation of the fatty acids. After 168 h of aging, bands at the range 1560-1575 cm^{-1}were observed, indicating the coordination of carboxylate groups tothe Cu(II) ions. SEM-EDS analysis shows that the 5h aged pigment has a non-uniform size distribution with a mean lateral particle size of 24.66 +- 7.12 µm and an increase in pigment agglomeration in the oil with increasing aging time reaching 46.44 +- 7.67 µm for the 720h aged paint. A remarkable difference is observed in the EPR spectra related to the Cu(II) signal in the samples after aging. For example, the original isotropic signal related to Cu(II) ion present in the malachite pigment changed to a rhombic signal after 5 h aging, whose simulation indicated the following EPR parameters: g_x= 2.08; g_y= 2.16 and g_z= 2.30 and A_x= 1.58 G, A_y= 66.48G and A_z= 163.08G. Together, the FTIR and EPR analyses showed that the copper(II) ion present in the malachite pigment undergoes a chemical reaction with the product of hydrolysis/oxidation of linseed oil, which results in pigment agglomeration and consequently, possible damages to the paint.

Acknowledgments: CNPq grant number 101518/2022-6

References:

[1] Cotte, M. et. al. Studies in conservation 62, 2 (2017)
[2] Seves, A. M. et al. Journal of Cultural Heritage 1, 315 (2000)
[3] Hills-Kimball, K. et. al. Inorganica Chimica Acta 529 (2022)

Characterization of Vale do Pindaré clay minerals forthe other industrial applications

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Four Maranhense clays obtained from brickworks of the small pole of Vale do Pindaré were characterized in a preliminary way by several methods aiming at their potential application in filters for water treatment or other applications. The mineralogy, plasticity, porosity, and interaction of clay with other components determine its suitability for use as filters[1]. The chemical composition of clay minerals was quantified by XRF. In both types of clay and evaluated as plastic (CIGD and CEGD) of a darker color and non-plastic (CIMG and CEMG) of a more yellowish color. A percentage of the largest oxides of silicon (Si02), aluminum (Al203), and iron (Fe203) is obtained, these being the largest non-plastic ones. Other oxides are found (Ti02, MgO, K20) with levels above 1%, which can give good resistance characteristics, as well as a high level of Al203 in all samples (>20%), which contributes to the application of clay as filters, thus improving their resistance mechanical, in addition to being an important refractory lining. XRD showed quartz as the most abundant phase in clays, while in more plastic clays its concentration decreased systematically with the increase of other phases found such as kaolinite, orthoclase and magnetite. The thermal behavior of the clays determined by TG showed a continuous loss of mass, divided into two events easily identified in the DTG, with peaks close to 50 °C and 550 °C. The first, associated with a loss of adsorbed water (2-3%) and a loss of mass at higher temperatures around 12.6%, for plastic clays and a loss of mass of 37% and 4.61% for the non plastic clays, respectively, is associated with loss of organic matter and dehydroxylation of kaolinite[2]. Future applications in the production of filters due to the presence of ions of interest such as potential mechanical characteristics.

Acknowledgements:

IFMA and IFPI

References:

[1] Ihekweme, G. O. et al. Heliyon 6, e03783 (2020)

[2] Tabet W.E., et.al. J. of. Material Eng, 30, (2018)

Characterization of ZnO thin films produced via an ultrafast and low-cost spray pyrolysis methodology

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Zinc oxide (ZnO) thin films have been explored for several applications, such as sensors, solar cells, photodetectors, LEDs, photocatalysts, and others [1-3]. In this work, a homemade system of spray pyrolysis was mounted and used to produce ZnO films, deposited on a glass substrate of 0.5mm thickness. The precursor solution was zinc nitrate ($Zn(NO_3)_2$) dissolved in de-ionized water, with concentration of 0.1M. During the deposition, the substrate temperature was controlled (280°C and 320°C were tested) and after the deposition the sample was annealed at 350°C for 1h, in a muffle-type furnace. The crystalline wurtzite (ICSD #26170) was the single phase observed in X-ray diffraction (XRD) results, with preferential orientation according to the substrate temperature.The SEM images revealed the uniform deposition of the films and the characteristic rod-like shape ofZnO particles. The optical characterization was performed in excitation and emission modes. ZnO presented a broadband emission between 400 nm and 800 nm, which was enhanced by the annealing of the samples. This result was possibly associated to thecrystallization and grain growth of the ZnO nanoparticles. In conclusion, this methodologypresented several advantages, such as low-cost, easy operation, good reproducibility, andcontrol of the properties. The surface homogeneity was comparable or even better to theresults previously obtained using dip or spin coating methods. In the next step of this work,ZnO doped with transition metals are currently being produced and characterized, to beexplored as photocatalists.

Acknowledgments:

This work has been supported by the brazilian funding agencies CNPq, CAPES, FINEP and FAPITEC. This research used facilities of the CMNano/UFS under proposal #069/2022.

References:

[1]Thamonwan Angkuratipakorn et. al., Ceramics International, 2023;

[2]Athorn Vora-ud et. al., Chemical Engineering Journal, 2023;

[3]M. Tamseel et.

Chemical, structural and photophysical investigation of the novel mixed-metal Eu^{3+}/Sb^{3+} layered rare earth hydroxides

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Layered rare earth hydroxides (LREHs) are a family of anion-exchangeable 2D inorganic materials that have been the object of interest for their new and adapted electrical, magnetic and optical properties in different fields of materials science [1]. Regarding the fluorescent properties of LREHs, the use of Sb^{3+}as sensitizing centers for Eu^{3+}has not yet been explored despite that the ns^2electronic configuration has resonant states with the Eu^{3+}energy levels and this can lead to efficient energy transfer processes (ET)[2]. In this work were obtained a series of LRHs heterometallic hydroxides ($LEu_{1-x}Sb_xH-Cl$ (x = 0.05, 0.10, 0.15, 0.20, 0,25, and 0.30) through hydrothermal treatment to examine the influence of the physicochemical properties and the ET resulting from the incorporation of Sb^{3+}. XRD for $LEu_{1-x}Sb_xH-Cl$ showed characteristic planes of the $Yb_8(OH)_{20}Cl_4{\cdot}nH_2O$ (orthorhombic crystal system; $Pca2_1$space group) corresponding to the crystallographic data. FT-IR spectra for $LEu_{1-x}Sb_xH-Cl$ revealed a band between 473-600 cm^{-1}related to M–O (M = Eu and/or Sb) and a signal between 3500-3750 cm^{-1}corresponding to the –OH band. EDS spectra show the presence of Sb and Eu for all $LEu_{1-x}Sb_xH-Cl$. Photoluminescence of $LEu_{1-x}Sb_xH-Cl$ reveals the characteristic *f-f* transitions $^5D_0\rightarrow{}^7F_{0-4}$from Eu^{3+}ion. On the other hand, excitation spectrum(λ_{Em}= 618 nm) revealed, in addition to the Eu^{3+} *f-f* intra-configurational transitions, a progressive increase of the band centered at 274 nm (Sb^{3+}: $^1S_0\rightarrow{}^3P_1$) suggesting a possible ET: $Sb^{3+}\rightarrow Eu^{3+}$. In short, it was performed for the first time a chemical, structural and photophysical spectroscopic study of $LEu_{1-x}Sb_xH-Cl$, revealing the influence on Sb3+ incorporation.

Acknowledgments: TecNano, CNPq, CAPES, FAPEAL

References:

[1] A. D. Yapryntsev, A. E. Baranchikov, V. K. Ivanov, Russ. Chem. Rev., 89, 629 - 666 (2020)

[2] Q.Yu, H. Zeng, Z. Liu, J. Ren, G. Chen, Z. Wang, L. Sun, J. Alloys Compd., 590, 92-95 (2014)

Comparative study of compression resistance and microstructure of geopolymeric cements with additionof blast furnace slag and steel blast

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Geopolymers are inorganic materials formed by a rich source of silicon and aluminum with an alkaline solution, by virtue to their strong bonds, these materials have chemical inertness, durability and high mechanical resistance. Blast furnace slag and steelmaking slag are industrial by-products that have a high calcium content in their structure, which promotes increased mechanical strength in the structure of geopolymers due to microstructural compaction. In this comparative work, the properties of compressive strength and microstructure of geopolymeric cements with addition of blast furnace slag and steelworks slag were studied. The specimens were tested after seven days of curing,and soon after the test, the fragments were taken for optical microscopy for microstructural evaluation. The cement with the addition of blast furnace slag obtained better mechanical performance, about 24 MPa, as well as a microstructure with few pores and more compact, when compared to the geopolymeric cement with the steel slag.

Acknowledgmet:

CAPES, CNPQ, PROPESP/UFPA, and the laboratories that comprise the composite materials research group of UFPA.

References:

[1]Cândido, V. S., da Silva, A. C. R., Simonassi, N. T., Lima, E. S., da Luz, F. S., Monteiro, S. N. (2018). Mechanical and microstructural characterization of geopolymeric concrete subjected to fatigue. Journal of materials research and technology, 7(4), 566-570.

[2] Davidovits, J. (1989). Geopolymers and geopolymeric materials. Journal of thermal analysis, 35, 429-441.

[3] Silva, A. C. R., Almeida, B. M., Lucas, M. M., Candido, V. S., da Cruz, K. S. P., Oliveira, M. S., ... Monteiro, S. N. (2022). Fatigue behavior of steel fiber reinforced geopolymer concrete. Case Studies in Construction Materials, 16, e00829.

Comparison between hydraulic and colloidal binders in the processing of refractory ceramic suspensions

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The direct casting of aqueous suspensions uses binders (hydraulic or colloidal) to set the structure, avoid drying shrinkage, and assure strength for demolding. Hydraulic binders are anhydrous oxides that react with water to harden the structure. The most used hydraulic binders in the refractories are calcium aluminate cement (CAC) and hydratable alumina (HA). Colloidals comprise nanod particles dispersed in a liquid phase that can be chemically gelled. For colloidal silica-bonded systems (CS) particles' gelling does not involve hydroxylation reactions. Due to this, the high-permeability structure generated reduces the pressurization by water vapor during drying. This work compared the physical properties of calcined alumina suspensions cast using HA or CS as binders after isothermal treatments (120-1600°C). Up to 900°C, CS-bonded samples showed the highest levels of elastic modulus. For the CS ones, the viscous sintering of SiO2 nanoparticles increased the rigidity, despite the higher total porosity levels. Above 900°C, HA-bonded samples became strongerdue to the early sintering, at the same time, in situ formation of mullite in CS-bonded-ones difficulted particles' densification due to its low density.

Acknowledgment: FAPESP (2010/19274-5), CNPq (147526/2021-3), CAPES (PNPD 88887.648527/2021-00), ALMATIS (Brazil and Germany), Nouryon SA (Brazil).

References:

[1] R. K. Iler, The colloid chemistry of silica and silicate, New York; Edit. Ithaca, 1979;

[2] R. Salomão et al., Advances in nanotechnology for refractories: When very small meets hot, heavy, and large; American Ceramic Society Bulletin, vol. 92, n.7, p. 22-28, 2013;

[3] R. Salomão et al., Hydratable Alumina-Bonded Suspensions: Evolution of Microstructure and Physical Properties During First Heating, InterCeram: I. C. R., vol. 66, n.7. S. F. W., 2017.

Conventional versus laser sintering of the high entropy oxide (Mg0,2Co0,2Ni0,2Cu0,2Zn0,2)O

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High entropy materials (HEM) can be obtained from several chemical elements, and commonly have a simple crystalline structure. In this group of materials, high configuration entropy can be obtained by equimolar mixing of constituents that hinder phase segregation at high temperatures, thus increasing the solubility of the components. In this work, high entropy oxide powders (Mg0,2Co0,2Ni0,2Cu0,2Zn0,2)O were synthed by the polymeric precursors. To sintering, the calcined powders were uniaxial compacted with a pressure of 80 kgf/cm³ in a cylindrical format, resulting in green ceramics measuring 4 mm in diameterby 1.0 mm in thickness and relative density of approximately 60%. The green ceramics were sintered using two methods: conventional sintering in electrical furnace at 1100 °C/2h, 1150 °C/2h and 1200 °C/2h; and by laser sintering, in which a CO2 laser is used as heating source, at different power density. The relative density of the sintered ceramics produced bythe both techniques was 85%. The samples were characterized by X-ray Diffraction (XRD),Scanning Electron Microscopy (SEM), Energy Dispersion X-ray Spectroscopy (EDS) and Impedance Spectroscopy (IS). XRD results confirm the formation of a single cubic crystalline phase (Fm-3m) [1] in all ceramics. Laser-sintered ceramics presented a smallergrain and lower surface porosity than conventionally sintered ceramics. EDS mapping images reveled a segregation of Cu at grain boundary associated with a relative decrease ofthe other ions [2].

Acknowledgements:
CNPq and CAPES.

References:

[1] YOON, B. et al., Scripta Materialia vol. 181, page 48-52 (2020).
[2] ZHAO, Y. et al., Journal of Materials Research and Technology vol. 22, page 3108-3114 (2023).

Detection of traces of the antibiotic Polymyxin B usinga fluorescent probe based on the FRET system of gold nanoparticle complexes coated with rhodamine B/sodium citrate

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Detection of traces of the antibiotic Polymyxin B using a fluorescent probe based on the FRET system of gold nanoparticle complexes coated with rhodamine B/sodium citrate

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Gold nanoparticles (AuNPs) are nanostructures with intrinsic optical properties attributed to localized surface plasmon resonance (LSPR). This phenomenon is provoked by the quantum confinement of electrons resulting in the appearance of bands (transverse and/or longitudinal) as a consequence of the excitation of AuNPs by electromagnetic radiation in the visible region [1]. Polymyxin B is a cationic polypeptide antibiotic used in last-resort therapies. The main objective of this work was to study the behavior of gold nanoparticles (AuNPs) to evaluate their potential as sensors for the quantification of traces of the antibiotic polymyxin B, using fluorescence as a parameter for the quantification of the contaminant [2]. The formation of pairs with the cationic dye Rhodamine B were based on fluorescence resonant energy transfer (FRET systems)-based processes for pairs of rhodamine B (donor) and acceptors of citrate capped gold nanoparticles (AuNPs) through incorporation progression of the analyte. Strong fluorescence activation was observed for polycationic species of polymyxin B whose detection limit is around 0.7 ppm, being the system satisfactory for detecting traces of polymyxin B.

Acknowledgment: FAPESB, FACEPE, CNPq, INFO-CAPES, FINEP.

References:
[1] X. Bai, Y. Wang, Z. Song, Y. Feng, Y. Chen, D. Zhang, L. Feng, International Journal of Molecular Sciences, 21(7), 2480 (2020).
[2] E.G.L. Oliveira, M.C.A. de Oliveira, Y. Xing, G. S. Macie, A. S. L. Gomes, H. P. de Oliveira, Results in Chemistry, 4, 100634 (2022)

Developing NaYF4 nanoparticles doped with rare earth and other ions as a proposal to improve the performance of solar cells

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The increasing demand for energy, coupled with growing concerns about the state of the environment and its impact, have intensified the need for renewable energy sources. As a result, research into the development and improvement of these sources is of great importance [1,2]. Most solar panels in use today are based on silicon (Si) and have limited absorption in the ultraviolet (UV) region, where the sun has strong emission. However, its highest absorption peak is in the near-infrared (NIR) region [3]. Given this consideration, the aim of this work is to develop mechanisms such as systems based on down-conversion and quantum-cutting to convert high-energy photons into lower-energy photons, to increase the efficiency of solar cells. For this purpose, many NaYF4 nanoparticles were synthed and doped with Ytterbium and different combinations of other rare earth ions, including Erbium, Neodymium, Cerium, Thulium, Manganese, and Gadolinium, all of them in their trivalent form. The idea is that these other rare earth ions absorb high-energy photons and, through non-radiative process, can transfer their energy to the ytterbium ions, which emit strongly in the IR region. As a result, we successfully obtained that, under UV excitation or by excitation with a white light source, characteristic emissions of Ytterbium ions were observed. The next step in the work will be to find the best combination of these ions and optimize their concentrations.

Development and characterization of model drilling fluid based on oxidized hexagonal boron nitride (h-BN-oxi)

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Ultra-deep water exploration has become necessary due to the rising demand for oil and difficulty in extracting it from onshore fields.[1] However, it poses challenges such as drilling through salt formations and environmental issues.[2]

The drilling fluid plays a crucial role in cooling the bit, transporting cuttings, and stabilizing rock formations. Moreover, the drilling process experiences elevated temperatures and pressures and one of the major challenges is to maintain the rheological properties of drilling fluids. To address these issues, companies have adopted nanotechnology-based solutions to develop fluids capable of operating at high pressure and temperature while ensuring material stability. One promising material is hexagonal boron nitride (h-BN), which is highly inert, non-toxic, and exhibits good thermal stability and hardness.[1] This work aims to develop oxidized hexagonal boron nitride (h-BN-oxi)/xanthan gum nanostructured model fluids to understand their rheological and thermal properties. Morphological and structural characterization of the model fluids was performed and related with the rheological studies.

Acknowledgements:

The CNPq (Project No. 406765/2022-7, 405665/2022-9, and 305109/2022-7), CAPES, FAPERJ, FAPESP (Grant No. 2012/50259-8, 2018/10910-8, 2017/17661-0, 2018/25210-1), and Petrobras.

Mackenzie Presbyterian University and MackPesquisa for the financial support.

References:

[1] SOARES, Y. C. F. et al. Multifunctional hexagonal boron nitride dispersions based in xanthan gum for use in drilling fluids. Journal of Petroleum Science and Engineering, v. 221, n. June 2022, 2023.

[2] ALCÁZAR-VARA, Luis Alberto; CORTÉS-MONROY, Ignacio Ramón. Drilling fluids for deepwater fields: An overview. Recent Insights in Petroleum Science and Engineering, v. 71, 2018.

Development mesoporous silica nanoparticles functionalized with chitosan

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Mesoporous silica nanoparticles have attracted interest from several areas of knowledge, including, but not limited, pharmaceuticals and cosmetics [1], due to their characteristics such biocompatibility, large surface area, enabling the functionalization and loading of bioactive molecules [2]. Thus, the objective of this work was to develop MCM-41 functionalized with chitosan (MCM-41@Ch) for drug release control. The synthesis of the MCM-41 type silica nanoparticles were developed by the sol-gel method using cetyltrimethylammonium bromide (CTAB) as a template, at mild temperature, involving hydrolysis and condensation reactions of tetraethyl orthosilicate (TEOS), precursor of silica. A 1% chitosan solution was prepared [3]. Subsequently, the 1% chitosan solution was used to functionalize MCM-41. Characterization tests carried out were FTIR spectra using a Shimadzu IR Prestige-21 spectrophotometer; XRD was made from a Shimadzu XRD-6000 x-ray diffractometer using CuKα radiation and λ=1.79 Å. TEM images were obtained using a transmission electron microscope Tecnai Spirit T12 analysis. XRD' result confirmed the formation of silica nanoparticles with hexagonal structure p6mm of ordenaly way, being a pore surrounded by six others pores. The TEM images of unmodified MCM-41 and the surface modifie nanoparticles (MCM-41@Ch) demonstrated that all of them showed uniform and spherical shape and have an average particle size. With the FTIR analysis was possible to investigate the functional groups and connections chemicals present in the samples. It wasconcluded that the MCM-41 particles reached a nanometric scale, as well as the functionalization with chitosan was achieved and that they can be used for the production of medicines and/or cosmeceuticals.

References:

[1]R.K.Kankala et al.,Advanced Materials vol.32(2020)
[2]L.F.A.M.Oliveira et al.,Drug Development and Industrial Pharmacy,v.46,p.1199-1208(2020)
[3]Q.Gan et al.,Journal of Materials Chemistry B, vol.3,p.2056-2066(2015)

Development of betadiketonates complexes luminescent and photosensitive for application as shotresidue marker

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Non-toxic ammunition (NTA) has been developed to protect the health of security agents as well as the environment itself. However, gunshot residues (GSR) generated by NTA ammunition has a certain level of difficulty when it comes to their unambiguous identification. To bypass this problem, the use of luminescent markers in ammunition has been proposed. These markers bring with them an advantage in the matter of identification, by allowing the convenience of visualizing them using only a portable ultraviolet lamp, facilitating the ability to collect directly on site for subsequent analysis in the laboratory.[1-2] In this work, the use of complex β-dicetonates as luminescent markers of ammunition was proposed. The complex β-dicetonates of Europium, $Eu(BTFA)_3bipy$, $Eu(BTFA)_3phen$ and $Eu(BTFA)_4$were synthetized, characterized and had their effectiveness as markers in assessed ammunition. The complexes containing 2.2'- bipyridine and 1.10-phenantroline were synthesized using a reflux system at 60 °C, while the $Eu(BTFA)_4$was synthetized using the same system, but at ambient temperature. Both of them are easily scalable for an industrial situation, the latter having the advantage of being able to be synthesized without the need for heating. The results of their characterizations before and after the shots showed that these three complexes are feasible as luminescent markers.

Acknowledgements:
Second place award at the 30th UFPE Scientific Initiation Congress

References:
[1] Lucena, M. A. M.; Ordoñez, C.; Weber, Forensic Science International 2017, 280 (November), 95-102.
[2] Arouca, A. M., Lucena, M. A. M. Journal of Forensic Sciences, 65(1), 67-72.

Development of binary and ternary oxides-based electrodes for electro-oxidation of organic pollutants in water

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Electrochemical technology is a great method for degradation of complexes and stables pollutants [1]. This work aimed the development of Fe, Co, Ti, Ni and Cu oxides-based electrodes, in binary and ternary compositions, for degradation of organic pollutants in water. The sun-yellow dye was the model pollutant. New materials were deposited into titanium plates and obtained by thermal decomposition at 400 ºC. These materials were analyzed as anode and a commercial DSA as cathode. Electrochemical characterization was performed in H_2SO_4 0.5 mol L^{-1}. The assays were realized for 2h in Na_2SO_4 0.1 mol L^{-1}, $j = 15$ mA cm^{-2}, at ambient temperature, under magnetic stirring and with no pH adjustment. For the electrodes analyzed, mean color removal rate were over 90% with good reproducibility. The open-circuit potential values obtained before and after the oxidation reactions presented low variation for the compositions Co-Fe, Ni-Ti-Fe and $Ti/Cu_{0.5}Fe_{0.5}O_2$. No correlation between the electroactive area and the electrocatalytic efficiency was observed. This suggests that the most crucial elements for the efficiency and stability are the generated oxide types and their crystalline structures. For the anode $Ti/Ni_{0.3}Ti_{0.2}Fe_{0.5}O_2$, the dye initial concentration decay data was best adjusted for the pseudo-second order, meanwhile, for the others, the pseudo-first order was a better fit. The results show that the materials are efficient and stable as anodes in the electro-oxidation. In addition, the small cost of the reagents needed to synthetize them, promote the possibility ofa simple, effective and accessible alternative to the wastewater treatment through electrochemical technology.

Acknowledgements

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Reference

[1] I. Sirés, E. Brillas, M. A. Oturan, M. A. Rodrigo, M. Panizza, Environ. Sci. Pollut. R. 21, 8336 (2014).

Development of superhydrophobic nanocomposite coatings based on electrodeposited nickel stearate and multi-walled carbon nanotubes

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A metal surface can exhibit low wettability through the formation of a hierarchical roughness structure in the micro/nanometer dimensions in association with a surface energy-reducing agent. When the contact and slip angles are ≥ 150° and ≤ 10°, respectively, the surface is characterized as superhydrophobic. [1,2] Superhydrophobicity gives the surface a self-cleaning effect, where water is highly repelled, protecting the metal substrate from direct contact with corrosive electrolytes. Among the ways to coat a material, electrodeposition stands out by presenting good versatility, low cost, relatively simple industrial application, and has shown to be an emerging technique to produce nanocomposite materials in several areas of materials engineering [2,3]. In this study, coatings containing nickel stearate and nickel stearate with multiple-walled carbon nanotubes were applied via electrodeposition in alcoholic solution on aeronautical aluminum alloy 7050. Contact angles of up to 160° (about twice as large as the uncoated substrate)and sliding angles of approximately 1° were obtained. Scanning Electron Microscopy (SEM)and Energy Dispersive Spectroscopy (EDS) analyses confirmed the presence of a hierarchical roughness structure resembling a cauliflower, responsible for conferring the superhydrophobic characteristic, formed by peaks characteristic of the elements Ni, Cl, C and O. The results of X-ray diffraction (XRD) showed the presence of peaks characteristic of nickel phases, proving the reduction of nickel ions on the aluminum surface.

Acknowledgments:

FINEP, CAPES, CNPq, FACEPE PROPESQI-UFPE, LBC-COMPOLAB/UFPE, LITPEG/UFPE and INTM/UFPE.

References:

[1] D. Jayakrishnan, Corrosion protection and control using nanomaterials. Woodhead Publishing., 86-125 (2012)

[2] Y. Lin, H. Chen, G. Wang and A. Liu, Coatings, v. 8, n. 6, 208 (2018)

[3] R. Miller, S. Hu, S.Weamie, S. Naame, D. Kiazolu, Journal of Materials Science and Chemical Engineering, v. 9, n. 4, 68-104 (2021)

Doxycycline and clay mineral as a hybridphotoprotective agent

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Antibiotics such as those from the tetracycline family, especially doxycycline, are widely used to treat various bacterial infections. However, their low stability is a challenge for the pharmaceutical industry, which can induce their degradation during the manufacturing process or storage. The formation of complexes stands out among the strategies studied to overcome the problem. This work investigated drug/clay complexes (DTC/VEEGUM and DTC/SEP) as photoprotector. The adsorption was performed using 2g of clay in 2g$L^{-1}$of the drug under agitation for one hour of reaction. Light stability tests were performed using a 125W lampless lamp as the UV radiation source for up to 200h. Photostability monitoring was performed using DRX, TEM, and BET. The XRD results showed changes in the crystallographic planes of the samples in the DTC/VEEGUM system before and after incorporation, but little change as a function of irradiation time, indicating its stability under UV light, as for the DTC/SEP system not. The TEM images corroborate the data observed in the XRD. In general, the specific surface area experimental data showed that the DTC load on the clays decreased the samples'' specific surface area and the total pore volume. This observation suggests that the pores were blocked upon embedding. It is concluded that DTC was successfully incorporated into the clay minerals under study from the evaluated systems. The DTC/VEEGUM interaction product showed more excellent stability to UV radiation, which may be due to its internal surface and the interlamellar space in its structure.

Effect of Ag nanoparticles on the electrochemical activity of a hybrid ormosil film with phosphomolibdicacid

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Hybrid thin films formed by polyoxometalates (POMs) immobilized in an organically modified silicate (ormosil) network have been used in applications as photo and electroactive devices [1,2]. The employment of metallic nanoparticles in films containing POMs can improve the electroactive activity of a hybrid film [3]. Very few experiments, however, show electrochemical properties of a hybrid ormosil film containing POMs and metallic nanoparticles, such as silver nanoparticles (Ag NPs). In this study, we investigated the electrochemical response of a hybrid ormosil film containing phosphomolybdic acid (HPMo), a Keggin type POM, and Ag NPs immobilized in an ormosil network formed by tetraethyl orthosilicate and 3-Glycidoxypropyltrimethoxy silane. The electrochemical properties were analyzed by cyclic voltammetry and differential pulse voltammetry. The results showed that in the hybrid ormosil film containing Ag NPs, the current density was 20,19% higher than the hybrid ormosil film without AgNPs. It was demonstrated that the hybrid ormosil film with AgNPs could be used to develop electrochemical devices and in future applications in this area.

Acknowledgments:

GVB would like to thank CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior) for providing his Ph.D. scholarship. We would like to thank Laboratório Nacionalde Nanotecnologia (LNNano) from Centro Nacional de Pesquisa em Energia e Materiais (CNPEM) for the preparation of gold electrodes used in this study.

References:

[1] P. H. Olívio, L. A. Correia, J. H. Paula, O. N. Oliveira Jr A. L. Souza. Materials Science and Engineering: B, v. 229, p. 13-19, 2018.

[2] M. de Oliveira Jr, A. L. de Souza, J. Schneider, U. P. Rodrigues-Filho. Chemistry of Materials, v. 23, n. 4, p. 953-963, 2011.

[3] S. Berbéc, S. Zoladek, P.J. Luesza, B. Palys. Journal of Electroanalytical Chemistry, v. 854, p. 113537, 2019.

Effect of cellulose nanofibrils on the self-aggregation of different bile salts

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Bile salts are surfactants produced by the liver, being important as drug delivery platforms [1]. Above the critical micellar concentration (cmc), they can form self-aggregates and have the structure altered by intermolecular interactions with substances [2] such as cellulose nanofibrils (CNF), creating procedures for obtaining rheological modifiers. In this work, CNF in the concentrations of 0.96, 2.40 and 4.80 g L-1 was mixed with sodium taurodeoxycholate (NaTDC) or sodium deoxycholate (NaDC) in order to evaluate the viscoelasticity of the systems by rheological measures. Flow curves of all systems showed that the apparent viscosity increased with the increment of CNF. Furthermore, the systems presented the phenomena of shear thinning followed by shear thickening, indicating the non-Newtonian behavior of fluids [3]. The viscoelasticity of the systems was confirmed by rheological curves in the oscillatory mode and the increment of CNF amount increases the viscoelasticity of the systems. But, CNF/NaDC systems presented more structured than CNF/NaTDC, since the elasticity modulus G’ increases. This suggests that, in the presenceof nanocellulose, NaDC self-aggregates are longer than bile salt with the taurine group, perhaps, due to the less negative charge in the NaDC structure. In short, CNF associated with both bile salts can form different structures depending on the proportion of the nanoparticles, modulating the rheological behavior of the systems. Then, different bile salts can be promising systems for rheological modifiers depending on the objective of application.

Acknowledgements:
CAPES, FAPESP (2022/02092-9; 2022/02089-8), Prof. Edvaldo Sabadini (IQ-UNICAMP) and Prof. Camila Rezende (IQ-UNICAMP).

References:
[1] S. Bashyal et al. **Journal of Controlled Release**, vol. 338, p. 644-661 (2021)
[2] B. Mukherjee et al. **RSC Advances**, vol. 6, n. 3, p. 1769-1781 (2016)
[3] C. W. Macosko. **Rheology**: Principles, Measurements and Applications, Wiley-VCH, New York (1994)

Effect of dispersing techniques on the particle distribution of nano Nb2O5 dispersion

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Understanding the behavior of nanoparticles (NPs) in aqueous suspensions is a crucial step for many applications. Compared to bulk materials, NPs exhibit distinct characteristics that must be evaluated due to their highly reactive nature [1]. After going through a milling ball process in order to break up agglomerates and achieve particles of uniform on a nanoscale [2], some dispersing method were tested on nano niobium pentoxide (Nano-Nb2O5). The hydrodynamic and distribution of the resulting dispersion were analyzed using dynamic light scattering (DLS) with different ultrasonication methodologies (Bath and Tip Ultrasonicator) for 5, 10, or 15 minutes, and with the use of a Polycarboxylate-based superplasticizer (SP) as a stabilizing agent. The dispersion s were also analyzed after 4 hours of sonication. The DLS results revealed that the application of sonication reduced the agglomeration of Nano-Nb2O5 for all durations tested. Interestingly, it was observed that the sonication process for 5 minutes yielded comparable results to those obtained with longer durations. However, after 4 hours, it is possible to observe sedimentation and agglomeration advance. The addition of the superplasticizer ensured the stability of the nanoparticle dispersion, preventing agglomeration again.

Acknowledgments:

This work was supported by FAPESQ, CNPq (Process 408905/2022-0 and 308353/2022-6), FACEPE (APQ-1251-3.03/22) and CAPES/Brazil (finance code 001).

References:

[1] Bhattacharjee, S. Journal of Controlled Release, 235, 337-351 (2016).

[2] Velo, M. M. A. C., Nascimento, T. R. L. Dental Materials, 35(11), e272-e285 (2019)

Effect of liquid environment to Bi-BTC MOFs obtainedby laser ablation

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High temperatures and pressures can be reached by cavitation bubbles from laser ablation in liquid environment (PLAL). The plasm and radical specimens raised from that process have been used to obtain metallics and oxides nanoparticles and to carry out chemical reactions in a non-conventional rout [1]. Matal organic frameworks (MOFs) is an important class of highly porous hybrid materials able to be obtained by laser ablation [2]. Recently, new MOFs based on bismuth and benzene-1,3,5-tricarboxyic acid (BTC) have been developed and laser ablation was applied to obtain Bi-BTC MOF in DMF/methanol liquid environment from bismuth target ablation [3,4]. Therefore, the effect of solvent has been less explored to investigate the MOFs structures obtained by laser ablation. This research presents results of bismuth MOFs obtained by laser ablation of powder bismuth oxide in three different solutions of BTC (DMF/methanol, DMF/ethanol and DMF/water) using a second harmonic (λ = 532 nm, 100 mJ) of a pulsed Nd:YAG laser. The scanning electron microscopy (SEM) presented sheet structures to Bi-BTC obtained in DMF/water and powder microstructure in DMF/methanol and DMF/ethanol. The samples were analyzed by FTIR, Raman, TGA and XRD, confirming different kinds of Bi-MOFs structures were found as a function of liquid environment.

Acknowledgments

This work was supported by: CNPq, CAPES and FACEPE.

References

[1] Campello, S.L., Gentil, G., Júnior, S.A., de Azevedo, W.M. (2015). Mater. Lett., 148, 200-203.

[2] Ataei, F., Dorranian, D., Motakef-Kazemi, N. (2020). J. Theor. Appl. Phys., 14, 1-8.

[3] Ye, F., Wei, Z.X., Song, J.F., Wu, X.H., Yue, P. (2017). Z. Anorg. Allg. Chem., 643(10), 669-674.

Electroactivity for nitrite of a layer-by-layer film with silver nanoparticles/phophomolibdic acid and poly(amidoamine)

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Hybrid layer-by-layer (LBL) films with polyoxometalates (POMs) have attracted much interest because of their wide range of applications as electroactivity for triazinic compounds [1,2]. Nitrite (NO2-) is generally used as food preservative and some studies have described its excessive consumption to the development of gastric cancer [3]. Hybrids in the form of LBL films formed by metallic nanoparticles (NPs) and POMs have not had their electroactive properties investigated in detail yet. In this study we aimed to investigate the electrochemical reactivity of films containing poly(amidoamine) (PAMAM) and silver NPs with phosphomolybdic acid (HPMo), a Keggin-type POM, toward nitrite. Cyclic voltammetry was used to explore the electrochemical properties of this hybrid film at pH 1.0. Results showed that nitrite was electroreduced by the film containing three bilayers of PAMAM, HPMo and silver NPs at -0,06 V vs. Ag/AgCl in the concentration range from 40 µmol L-1 to 234 µmol L-1. The LBL film containing PAMAM, HPMo and silver NPs showed higher sensitivity towards nitrite in comparison to the film without silver NPs with values of 5,20x10-4 µA cm-2 µmol-1 Land 1,07x10-4 µA cm-2 µmol-1, respectively. Our experiments demonstrate that this LbL film could be employed in systems for food and health applications.

Acknowledgements:

GVB would like to thank CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior) for providing his Ph.D. scholarship. The authors are grateful to LNNano (Laboratório Nacional de Nanotecnologia) from CNPEM (Centro Nacional de Pesquisa em Energia e Materiais) for the preparation of gold electrodes.

References:

[1] P.H.P. Olívio et al., Materials Science and Engineering B, vol. 229, page 13-19 (2018).

[2] A.L. Souza et al., RSC Advances, vol. 4, 29612 (2014).

[3] F.X. Zhang et al., Medical Science Monitor, vol. 25, page 1788-1799 (2019).

Enhanced Efficiency at Maximum Power in aFock–Darwin Model Quantum Dot Engine

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The study of quantum thermal machines, devices such as quantum engines and refrigerators that operate on working mediums composed of quantum systems, has proved to be a fruitful research area in the last decade [1]. These efforts have been focused principally on the study of the different thermodynamic cycles, such as the Otto and the Stirling cycles, in different regimes [2] and on the use of different quantum working substances such as spins, quantum dots [3], and quantum harmonic oscillators. Recent work has also examined the role that quantum properties, such as quantum coherence and quantum correlations, play in the performance of quantum thermal machines [4]. A primary feature of interest of quantum thermal machines is their potential to surpass the performance of their classical counterparts, which opens the door to a new generation of highly efficient quantum engines and refrigerators for application in emerging quantum technologies. In this work, we study the performance of an endoreversible magnetic Otto cycle with a working substance composed of a single quantum dot described using the well-known Fock-Darwin model. We find that tuning the intensity of the parabolic trap (geometrical confinement) impacts the proposed cycle’s performance, quantified by the power, work, efficiency, and parameter region where the cycle operates as an engine. We demonstrate that a parameter region exists where the efficiency at maximum output power exceeds the Curzon-Ahlborn efficiency, the efficiency at maximum power achieved by a classical working substance.

Evaluation of materials applied in the removal ofsodium diclofenac concentration.

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Diclofenac sodium is a widely used non-steroidal anti-inflammatory drug that can be found in water from rivers, lakes, and other sources. Therefore, the evaluation of materials applied in the removal of sodium diclofenac concentration involves both the study of different techniques and materials and the determination of removal efficiency, kinetics of adsorption or degradation, regeneration capacity and stability of the material under environmental conditions. Among the techniques used, adsorption on solid materials, advanced oxidation and heterogeneous photocatalysis stand out. In adsorption, the solid materials used have a high adsorption capacity (better put how the adsorption takes place) and can be reused after regeneration. In advanced oxidation, the drug is degraded through chemical reactions that use oxidants such as ozone, hydrogen peroxide and free radicals. In heterogeneous photocatalysis, drug degradation occurs through exposure to light, in the presence of catalysts such as titanium dioxide. Materials are chosen based on the conditions of use, the characteristics of the drug to be removed and material properties, such as, for example, adsorption capacity, stability and ease of regeneration. Therefore, some of the materials used in the removal of diclofenac sodium are activated carbon, zeolites, silicas, polymers and nanomaterials. Therefore, these studies are fundamental for the development of efficient and sustainable processes for the removal of sodium diclofenac and other contaminants emerging from water.

Evaluation of the adsorptive potential to atrazine of demolition and construction waste from State of Alagoas

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It is estimated that the civil construction industry in Brazil is responsible for generating 2.65 billion tons of total waste per year [1]. In addition, due to excessive use in agriculture, several bodies of water are now registering the presence of atrazine, an herbicide with the potential to be toxic to fauna and humans. [2]. Thus, this work evaluated the feasibility of applying construction and demolition waste (CDW) from Maceió/AL - Brazil as adsorbents in the removal of atrazine from aqueous matrices. The material was standardized to 0.59 mm, characterized, and tested for affinity with 10 ppm atrazine solutions at pHs 3, 7, and 11 in batch system. To evaluate the potential for industrial use, the material was applied in a fixed bed column (Di = 1.27cm, L = 5.0 cm) under a flow rate of 29.41 mL/min. It was noted that the material presented functional groups characteristic of clay mineral compounds, with absorption bands indicating O-Al, O-Fe, CaO and CaCO3. Little evidence of organic functionalization was identified. Superficially, the RDCs were not very porous and highly rough, and the batch affinity tests indicated that the material has a better adsorption potential at acidic pH, with a removal of 47%. In the fixed bed tests, percentages of removal in the range of 39.5% were observed, suggesting their applicability in the separation of a potentially present pollutant in water bodies and industrial effluents. Besides, the study opens the possibilities of converting heterogeneous waste like CDWs to full adsorbents.

Acknowledgements:
The authors thank Braskem S.A. for financial support.

References:

[1] TAM, V. W. Y; SOOMRO, M.; EVANGELISTA, A.C.J. A review of recycled aggregate in concrete applications (2000-2017), Const. Build. Mat., 172, pp. 272-292, 2018.

[2] CORREIA, F. V., LANGENBACH, T., CAMPOS, T. M. Avaliação do transporte de atrazinaem solos sob diferentes condições de manejo agrícola. Revista Brasileira de Ciência Do Solo,34(2), 525-534. (2010).

Evaluation of the Eu(btfa)$_3$bipy β-Diketonate Complexas an Ammunition Luminescent Marker for Detection of Gunshot Residues

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Non-Toxic Ammunition (NTA) was developed to protect the health of public safety agents and the environment. However, the methodologies employed for the identification of gunshot residues (GSR) have become inefficient when using this type of ammunition. In order to solve this problem, the implementation of luminescent markers in ammunition was presented as an alternative. These markers are known for allowing the visualization of residues at the crime scene using only an ultraviolet (UV) lamp, facilitating the visualization, collection and subsequent analysis of residues in the laboratory [1-3]. In addition, the waste generated can be characterized by various non-destructive analytical techniques available in the main expertise centers in Brazil [2]. The objective of this work is to evaluate the efficiency of the lanthanide β-diketonate complex (Eu(btfa)$_3$bipy) as a luminescent marker for shot residues. The results show that the Eu(btfa)$_3$bipy complex is an efficient luminescent and chemical marker, and its characterization can be applied to routine forensics.

Keywords: β-diketonates, lanthanides, luminescent marker, gunshot residues (GSR), forensic investigation.

Acknowledgements:

To CAPES, for the financial support, to the BSTR Laboratory of UFPE and to the professors for the support and past scientific knowledge.

References:

[1] Lucena, M. A. M.; Arouca, A. M.; Talhavini, M.; Alves-Júnior, s.; Weber, I. T. Ammunition encoding by means of co-doped luminescent markers. Microchemical Journal, v. 145, p. 539-546, 2019.

[2] Lucena, M. A. M.; De Sá, G. F.; Rodrigues, M. O.; Alves Júnior, S.; Talhavini, M.; Weber, I. T. Znal2o4-based luminescent marker for gunshot residue identification and ammunition traceability. Analytical methods, v.5 n.3, p.705-709. 2013.

[3] Weber, I. T.; Melo, A. J. G.; Lucena, M. A. M.; Rodrigues, M. O.; Júnior, S. A. Processo de obtenção de munição luminescente e processo de detecção de resíduos de tiro. 2010.

Evaluation of the influence of different parameters onthe fatigue behavior of geopolymeric concrete

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In the present work, flexural and fatigue tensile tests were carried out to evaluate the mechanical properties of Geopolymer Cement Concrete (GCC), which had characteristics compared with Portland Cement Concrete (PCC) [1], [2], [3]. The fatigue study was carried out by means of dynamic tests, in both types of concrete, evaluating the effects of different ages (1, 7 and 28 days), variation of minimum applied stress levels ($R=(\sigma min/fct,m)$) of 0.07, 0.20 and 0.40 and two percentages of steel fiber addition (0.5% and 1%). The fatigue results at different ages showed that the CCG has a better performance at all evaluated ages. The values found in the CCG were 13%, 96% and 24% higher than the values found in the CCP, at the ages of 1, 7 and 28 days, respectively. Regarding the minimum applied voltage, the average of the results showed a better performance of the CCG in relation to the CCP, presenting values 50% higher. Regarding the study with the addition of metallic fibers, the statistical values showed that the CCP and CCG had similar values. However, when observing the average values, it is possible to see the best results of the CCG in relation to the CCP, in the two percentages evaluated. In the microstructural analysis of the concretes it was possible to observe the better adhesion matrix-aggregate and matrix-fiber in the microstructure of the CCG, in comparison with the microstructure of the CCP, by virtue the absence of formation of crystalline phases in the matrix-aggregate and matrix-metal fiber interfacial region.

Acknowledgment:
CAPES and CNPq for financial support, Companhia Siderúrgica de Tubarão for the EGAF donation, and IME's Laboratory of Construction Materials and Soils.

References:

[1] J, Davidovits. Geopolymer Cement. A review, Geopolymer Institute Library. Jan. 2013.
[2] Zhang B, Wu K. Residual fatigue strength and stiffness ofordinary concrete under bending. Cement Concrete Res1997;27:115-26.
[3] Miner MA. Cumulative damage in fatigue. ASME 1945.

Evaluation of the Lotus and Salvinia Effects on Biomimmetic Coatings obtained in Aluminum 5052

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Various natural systems possess properties of great interest to industry and academia, such as self-regeneration, camouflage, high adhesion etc. Biomimicry consists in transferring those biological strategies to products, technologies, and other fields to solve practical problems. Certain plants, such as Nelumbo Nucifera or Salvinia Molesta, exhibit superhydrophobicity, which is the ability to repel water, inspiring the production of new materials with properties such as anti-corrosion, anti-biofouling, and self-cleaning.[1,2] In this study, aluminum 5052 substrates were modified to achieve and characterize superhydrophobic surfaces while evaluating the Lotus and Salvinia Effects on the properties of the obtained coatings. The AA5052 samples were modified by means of chemical attack using HCl (2M), formation of a layered double-hydroxide (LDH) film in a solution of zinc nitrate with ammonium hydroxide (1M), and finally, reduction of surface energy using lauric acid solution (0.028M). Morphological and chemical characterizations by SEM and EDS, potentiodynamic polarization and visual tests were conducted. The coatings exhibited self-cleaning behavior (Lotus Effect) as well as the formation of an air pocket when immersed in water (Salvinia Effect) due to their high-water repellency, with a contact angle of 162°. The morphology of the coating showed micrometric flowers with hexagonal nanometric petals that allow air trapping according to the Cassie-Baxter model of wettability. Through Tafel parameters, the improvement in anticorrosive behavior was confirmed, with a corrosion resistance efficiency of 81.24%.

Acknowledgements:
FINEP, CAPES, CNPq, FACEPE, PROPESQI-UFPE, LBC-COMPOLAB/UFPE, LITPEG/UFPE and INTM/UFPE.

References:

[1] JAMEI, Elmira; VRCELJ, Zora. Biomimicry and the built environment, learning from nature’s solutions. Applied Sciences, v. 11, n. 16, p. 7514 (2021)
[2] VON BAEYER, Hans Christian. The lotus effect. The Sciences, v. 40, n. 1, p. 12-12 (2000)

Evaluation of the structural, textural, and magnetic properties of NiZnMn ferrite synthesized via combustion reaction

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NiZn ferrite is an easily obtainable and relatively inexpensive material, making it a viable choice for many electronic applications, such as transformer and inductor core material, to suppress electromagnetic interference (EMI) and to reduce noise in electronic circuits [1,2]. Mn doping can be applied as a specific technique to optimize this NiZn ferrite in terms of its magnetic properties and thermal stability. In this study, ferrite Ni0.5-xZn0.5-xMn2xFe2O4 (x=0.1 and 0.15), that is, NiZn doped with 0.2 and 0.3 mol of Mn, was synthesized by combustion reaction in pilot scale, and sintered at 1200°C/1h. Afterwards, the samples had their structural, textural and magnetic properties evaluated. In this sense, the samples were analyzed by XRD, nitrogen adsorption/desorption (BET/BJH) and magnetic measurements (VSM). The results confirmed the monophasic formation of the studied nanoferrites, with crystallite s ranging from 33 to 57 nm and crystallinity from 45 to 82%, thus confirming the efficiency of the synthesis by combustion reaction. The samples showed particle ranging from 0.02 to 0.16 nm and surface area from 5 to 64 m2/g, where the larger the particle , the smaller the surface area. It was observed that both the introduction of manganese and the heat treatment favored the increase in the saturation magnetization of the samples, whose values ranged from 22 to 86 emu/g. Therefore, these results may be relevant for the development of large-scale NiZnMn ferrites with suitable properties for various applications.

Acknowledgements:
The authors thank to CNPq and CAPES by the financial support.

References:

[1] X. Zhou, J. Wang, D. Yao, Journal of Alloys and Compounds, v. 935, p. 167777 (2023).
[2] E. Leal, S.T. Basílio, J. Dantas, P. Richa, R.C. Lima, R.H.G.A. Kiminami, A.C.F.M. Costa. Arabian Journal of Chemistry, v. 13, p. 8100-8118 (2020).

Exploring the Potential of P-type Schwarzites for Selective CO_2 Adsorption: A Molecular Dynamics and GCMC Study

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Transportation and storage of CO_2 has become increasingly important due to the ever growing concern over anthropogenic greenhouse gas emissions [1]. In this work, we explored the potential of Schwarzites, three dimensional crystalline carbon structures initially proposed in 1991 by Mackay and Terrones [2], for the selective adsorption of CO_2. Specifically, we focused on the P family of Schwarzites, including the P8-1, P8-3 and P8-7, which have already demonstrated promising properties for CO_2 adsorption in previous studies. Using molecular dynamics and Grand Canonical Monte Carlo (GCMC) simulations, through the widely used Lammps tool [3], we looked at the adsorption of CO_2, as well as N_2 and O_2, at pressures ranging from 0.01 to 50 bars and a temperature of 300K. Our results showed the P8-3 to be generally the most interesting, presenting high capacity and selectivity for CO_2 adsorption, demonstrating the potential of Schwarzites as efficient materials for carbon capture and storage.

ACKNOWLEDGEMENTS:

We thank the Physics Department at Federal University of Parana for computational and financial support. We also thank National Council for Scientific and Technological Development (CNPq) for the financial support.

REFERENCES:

[1] N. N. C. for Environmental Information, “Monthly global climatereport for annual 2022”, 2023.

[2] A. L. MACKAY and H. TERRONES, Nature vol. 352, p. 762, 1991.

[3] A. P. Thompson, H. M. Aktulga, R. Berger, D. S. Bolintineanu, W. M. Brown, P. S. Crozier, P. J. in’t Veld, A. Kohlmeyer, S. G. Moore, T. D. Nguyen, R. Shan, M. J. Stevens, J. Tranchida, C. Trott, andS. J. Plimpton, “Lammps - a flexible simulation tool for particle-based materials modeling at the atomic, meso, and continuum scales”, Computer Physics Communications, vol. 271, 2 2022.

FeOCoAg nanocomposite synthed by catalyticcombustion

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In this work we used the synthesis process by combustion [1] in a digital muffle furnace (SolidSteel-SSFM (100°C-1200°C) with temperature control and stable atmosphere. The following reagents were used in this work: Ferric chloride ($FeCl3.6H2O$) P.A. , Cobalt acetate ($Co(C2H3O2)2.4H2O$) P.A., silver nitrate ($AgNO3$) P.A., and the catalyst fuels urea ($CH4N2O$) P.A. and citric acid ($C6H8O7.H2O$). The masses were made in the proportion of 1:1:1:3 respectively. After being homogenized in a pestle, they were inserted into a crucible and underwent the catalyst activation process, initially at a temperature of 120°C for 20 minutes four times. After this step, calcination was performed at 150°C once for 10 minutesand at 200°C for 20 minutes. The samples after synthesis were characterized by XRD, EDS,FTIR, UV-Vis-NIR, TEM and scanning microscopy. Optical microscopy observations were also performed. XRD measurements showed that the structure (FeOCoAg) was evidencedusing the Rietveld refinement method. The fluorescence measurements showed the atomic constituents with their respective energies for Fe, O, Co and Ag. FTIR measurements showed the presence of metallic compounds in the low wavenumber region. The UV-Vis-NIR measurements showed that the GAP energies present energy around 3.8 eV. The TEM measurements showed the morphology of the sample and after making the lognormal adjustments of the diameter distribution, it showed a mean diameter of 15 nm. Scanning microscopy showed that the morphology of the nanoparticles has spheroid surfaces.

Acknowledgements:

To Professor Alessandro Martins for the EDS and FTIR measurements, o IFRO, UNIR, LaBMic-UFG.

References:

[[1] Jonh J. Moore H. J. Feng, Procress in Materials Science Vol. 39, pp 243-273, 1995, Pergamon.

Fluorescent film based on carbon dots derived from lemon peel extract applied in fluorescent thermometry

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The development of fluorescent nanothermometers has been extensively studied due to their non-invasive measurement, wide temperature range and high resolution [1]. In this sense, carbon dots are excellent candidates to act as fluorescent nanothermometer due to excellent optical properties with tunable fluorescence, biocompatibility, photostability, and high dispersion in water [2]. In this work, a fluorescent film based on carbon dots (CDL-F) derived from lemon peel extract embedded in the polymer (PVA; poly (vinyl alcohol)) has been applied as luminescent nanothermometers. TEM micrographs for CDL revealed a particle distribution of 2.47 ± 0.47 nm and regular spherical morphology. FTIR spectrum showed the presence of the characteristic organic functional groups from the lemon extract: OH (3440 cm^{-1}), C–H (2930 cm^{-1}), C=O (1720 cm^{-1}), C=C (1400 cm^{-1}), and C–N (1200 cm^{-1}). Furthermore, the CDL showed absorption bands related to $\pi \rightarrow \pi^*$ (C=C) and $n \rightarrow \pi^*$(C=O/C-N) transitions. CDL exhibited excitation-wavelength-independent emission with maximum intensity at 446 nm (λ_{exc}= 336 nm). On the other hand, CDL-F showed excellent transparency, flexibility, and fluorescence in the blue region, which was similar to CDL in colloidal suspension. CDL-F also shows temperature-dependent fluorescence intensities between 298-333 K with a maximum relative thermal sensitivity of 2.69 % K^{-1}at 333 K and was stable over five cycles. In addition, MTT assay using a human non-tumoral skin fibroblast cell line (HFF-1) showed viability greater than 85 %. Finally, the recent results obtained indicate that CDL-F presents itself as a promising platform for luminescence thermometry applications.

Acknowledgments: CNPq, CAPES, UFAL, FAPEAL

References

[1] N. Jurga et al., Journal of Colloid and Interface Science vol. 606, 1421-1434 (2022)
[2] L.J. Mohammed e K.M. Omer, Nanoscale Research Letters vol. 15, 182 (2020)

Gelcasting colloidal silica-sodium alginatemacrospheres

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Ceramic macrospheres (0.1 1 mm) can be used as proppants for shale gas extraction, elements for milling, polishing, and fluidized bed process, and loose-fill insulation. Gelcasting is a method in which a suspension of ceramic particles containing a dissolved cross-linkable polymer is dripped and gelated in contact with an ionotropic coagulating bath. The beads produced show great control in chemical composition, geometry dimension, and concentration of defects. This study evaluated the influence of production parameters on the porosity, size, and shape of gelcast colloidal silica spheres. Mixtures of water, colloidal silica (10 35 wt% solids), and alginate (3 wt%) were dripped into a Ca(NO3)2 aqueous solution using needles of different diameters (0.7 1.6 mm). Their size distribution was determined using optical and scanning electron microscopy and porosity was measured by Archimedes Principle. The gelcasting technique enabled the production of spheres using colloidal particles of tunable sizes and porosity levels. Smaller beads were produced using thinner needles and lower solid contents at the same time all samples presented similar total porosity levels. Samples with 10 wt% solids showed non-uniform shrinkage, which did not allow the formation of perfect spheres after drying; those with 35 wt% showed very high viscosity and elastic behavior during extrusion, hindering the formation of beads at this concentration. Using 30 wt% solids content produced denser spheres (40 % porosity), with lower levels of shrinkage during drying, and presenting a more regular morphology and narrow diameter distribution. Such behavior was also observed reducing the needle’s diameter.

Green Synthesis of Metal-Based Nanoparticles Using Extracts from Mastic, Garlic, and Oregano.

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Green synthesis is a more sustainable approach to nanoparticle synthesis as it avoids the use of toxic chemicals and reduces waste. The extracts of plants contain natural compounds that can act as reducing agents and stabilize the nanoparticles, preventing them from aggregating. This synthesis process involves mixing the plant extracts with metal salts in a solution. The reducing agents in the plant extracts convert the metal ions into metal nanoparticles, and the resulting solution is then purified and characterized to confirm the , shape, and stability of the nanoparticles. Mastic is rich in terpenes and phenolic compounds. Garlic contains sulfur compounds, flavonoids, and other bioactive compounds. Oregano contains phenolic acids and flavonoids. Using the advantages offered by the green synthesis and the mentioned extracts, the metallic nanoparticles were synthed according to Otero et. al. (2022), and later identified in this study. The morphological, structural, and optical properties were comprehensively investigated through scanning electron microscopy, infrared spectroscopy, and optical absorption techniques. The results confirmed the successful formation of nanoparticles. To further evaluate the microbial activity of the synthed nanoparticles, a series of tests were performed.Therefore,metal-based nanoparticles synthed via green synthesis using mastic, garlic, and oregano extracts have shown promising results and can be used in various applications, such as antimicrobial agents, drug delivery, and catalysis. They offer a sustainable and eco-friendly alternative to traditional chemical synthesis methods.

ACKNOWLEDGEMENTS: CNPQ, FAPEAL and CAPES.

References: OTERO, M. J. B. et al. Chromatographic analysis of phytochemicals in the peel of Musa paradisiaca to synthe silver nanoparticles. Rev.fac.ing.univ. Antioquia nº 103 Medellín Apr./June 2022.

Hybrid Nanoplatforms based on Carbon Polymer Dotsand Halloysite

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Fluorescent nanomaterials have been extensively explored in recent years due to their promising applications in several fields of material science. Carbon polymer dots (CPD) show intense fluorescence emission, biocompatibility, water solubility and prominent optical properties [1], have gained significant research interest owing to their exceptional features. On the other hand, clay minerals have been identified as a promising candidate to compose versatile and multifunctional hybrid nanoplatforms for diverse applications. In this sense, Halloysite (HAL) displays unique characteristics that combine excellent mechanical properties, biocompatibility and versatile surface chemistry, being a good choice of inorganic counterpart in the elaboration of hybrid nanostructures [2]. Based on these premises, the present study aimed to develop a novel hybrid system based on CPD and the HAL clay mineral. The hybrid material was characterized by several instrumental techniques. In addition to intense blue fluorescence of the hybrid material when excited under UV light (365nm), the FTIR spectra suggested interactions between the main functional groups of CPD with halloysite surface. XRD technique revealed a slight of the peaks of HAL to low angles, which could be an indication of a possible increase of the amorphous phase in the system. In the thermal analysis from DSC, an improvement in the thermal stability of the hybrid compound was observed in comparison with the CPD alone, at the same time that these materials show interesting textural properties, which were evaluated by BET measurements. These materials can provide several applications such as attractive nanoplatforms for active molecules in drug delivery systems, optical and electronics or food sector.

Acknowledgments:
FAPEMA, UFMA.

References:

[1] C.Xia, S.Zhu, T.Feng,M.Yang,B.Yang, Advanced Science, 6, 1901316 (2019)
[2] N. Danyliuk, J.Tomaszewska, T.Tatarchuk, Journal of molecular liquids, 309, 113077 (2020)

Implementation of CQDs as green tracer fluids in petroleum reservoir

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The present work proposed a simplified procedure aimed at manufacturing environmentally safe, non-toxic Carbon Quantum Dots (CQDs) [1]. Despite the current worldwide expansion of nanotechnology, in the oil and gas market this area is yet incipient, although with promising perspectives. Biomass or bioresidues for the production of CQDs are alternatives to reduce the environmental and economic impacts, although these precursors are commonly misused or incorrectly discarded. Orange (Citrus sinensis) peels, for instance, areresidues composed of both soluble and insoluble carbohydrates, with high flavonoid contentand minerals, and, therefore, are excellent sources of carbon. Hence, a process to produceCQD from C. sinensis was applied via hydrothermal method, a facile, single-step and low- cost methodology, at constant temperature (180 ºC) for 8 hours, for the destination of tracerfluids in oil reservoir. A preliminary characterization of the material was performed throughcontact angle test (optical tensiometer) in carbonate rock and UV-Vis spectrophotometry, aiming to determine the stability of the CQD. The contact angle analysis, using pure dolomite rock (without petroleum), showed an angle inferior to 90º, whereas the experimentusing the oil-soaked rock the angle was higher than 90º, although this angle decreases withtime (30 min) due to the fluid-rock interaction, indicating that stone becomes wettable bythe fluid. In the UV-Vis analysis of the CQDs, only one absorption peak was observed at 272nm, which is within the expected range, and it remained stable for 8 days.

Acknowledgements:

All authors thank CAPES for the financial support.

Referências:

[1] John, B.K., Abraham, T. Mathew, B. A Review on Characterization Techniques for Carbon Quantum Dots and Their Applications in Agrochemical Residue Detection. J Fluoresc 32, 449-471 (2022). https://doi.org/10.1007/s10895-021-02852-8

Improvement of mechanicals gypsum properties by additioning different products.

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The Gypsum, Calcium sulfate hemihydrate (CaSO_4 0,5H_2 O), is a material employed in constructions to make lines, plasterboards and bricks. The Pernambuco state is the biggest producer of gypsum in Brazil, and Araripe region represents 80% of the national production [1]. Gypsum ß is the type of gypsum used the most in building, considering its easy recrystallization and low cost. Although such qualities, it still need to be more studied in order to improve its mechanical properties, as its poor resistance to compression or its low hardness. Therefore, this research, utilizing gypsum ß from Araripe region, aims to provide viable solutions to that problem. First, it was characterized the gypsum slurry; then, it was produced 39 specimens to test the mechanicals characterization (hardness and compressive strength), adding three different products (A, B and C) and different ratios (5%, 10%, 20% and 30%). The test was conducted according to NBR 12.128/2019 and NBR 12.129/2019, respectively. The results show enhancement of gypsum hardness and changes in its compressive strength.

Acknowledgement

References

[1] Agencia Nacional de Mineração. Sumário Mineral Brasileiro. Gipsita. pag.1 2018.

Improving the persistent luminescence properties ofthe BaAl2O4:Eu2+, RE3+laser-sintered ceramics by double co-doping

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This work reports the influence of co-doping on the luminescence and persistent luminescence (PersL) properties of BaAl2O4 ceramics. The samples were doped with Eu and double co-doped with different rare earths (Dy, Er, Nd, Pr, and Tm) and laser-sintered in open atmosphere at a power density of 1.6 W/mm2 . All sintered ceramics present the hexagonal BaAl2O4 phase, homogeneous microstructure. It was observed a photoluminescence emission band centered at 495 nm, which is due to the 4f6 5d1 → 4f7 Eu2+ transition in the BaAl2O4 matrix, indicating that Eu valence reduction has been achieved during laser sintering, even though the whole process was performed without atmosphere control. The PersL emission was monitored by 15 min after charging at 345 nm for 3 min. It is observed that the enhancement of the decay time and PersL intensity when the sample was double co-doped with the two best single co-dopants ions (Nd and Dy). These results suggest that the two co-dopants interact by modifying the traps configuration in the BaAl2O4 matrix being different from the simple sum of the PersL emission of the two co-dopants individually. Acknowledgements: CNPq and CAPES.

Influence of experimental parameters on the green synthesis of Y2O3:Eu3+

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Methodologies called Green chemistry have been highlighted in the scientific community due to the appeal for the reduction of toxic and polluting substances. In this sense, the present work aims to perform the Y2O3 particle synthesis doped with Europium ions (Eu3+), using a green route that employs aloe vera extract. Three different procedures were used to process the precursor solution: sonochemical, microwave-assisted hydrothermal,and magnetic stirring. For all methods, the Y2O3 precursor solution was prepared as follows: Y(NO3)3 and Eu (NO3)3 as reagents, and the gel extracted from aloe vera, with a 30%w in aqueous solution. The Y concentration in the precursor solution was 0.1 M, and the Eu ions were used in a concentration of 5mol% respective to the Y content. The sonochemical route and magnetic stirring were used for 6 hours at room temperature and the microwave-assisted hydrothermal route for 2 hours at 90ºC. After the initial processing,all the samples were calcined at 700ºC for 7 hours. For comparison, the synthesis of Y2O3:Eu3+ without aloe vera extract was also performed. The Y2O3:Eu3+ particles were characterized using XRD, SEM, and PL techniques. As a structural result, through all routes, the crystalline cubic Y2O3 is obtained. From the SEM analyses, it is also observed that the particles are organized in clusters of a few micrometers, which are smaller when synthed with aloe vera gel. In the luminescence, the characteristic response of the Eu3+ was noticed in the emission and excitation analyses, and interesting results for promissory application in solar cells were observed.

Acknowledgments: This work has been supported by the Brazilian funding agencies CNPq, CAPES, FINEP and FAPITEC. This research used facilities of the CMNano/UFS under proposal #080/2023.

Influence of nanocellulose on the sodium deoxycholate self-aggregation

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Sodium deoxycholate (NaDC) is a bile salt that above the critical micellar concentration (cmc) form aggregates spontaneously in ordered and/or disordered phases. The structure of these self-aggregates can be altered in the presence of certain molecules [1], such as cellulose nanocrystals (CNC) or nanofibrils (CNF), leading to changes in the rheological behavior of systems. It's strategic to study how CNC and CNF influence on the autoaggregation of NaDC, since there're few works that studied the aggregation of bile salts and nanocellulose [2]. Here, the rheology of NaDC (50 or 100 mM) self-assembled systems in aqueous medium was evaluated about CNC or CNF influence in the concentrations of 0.96, 2.40 and 4.80 g L^{-1}. Flow curves of all systems showed that the apparent viscosity increased with the increment of CNC or CNF concentration and presented the non-Newtonian behavior of fluids [3]. Dynamic assay in the oscillatory mode showed that NaDC 50 mM systems with CNC or CNF are much more structured and viscoelastic than CNC or CNF/NaDC 100 mM systems, since the elasticity modulus G' is greater than G" in the first case. This phenomenon suggests that NaDC 50 mM systems could possibly be gels. Also, CNF/NaDC aggregates for both surfactant concentrations were more structured and longer than CNC/NaDC systems, since G' increased. Summing up, the morphology and viscoelasticity of these self-aggregates can be altered with the proportion of nanocellulose and, mainly, of surfactant, being promising for new systems of controlled release.

Acknowledgements:

CAPES, FAPESP (2022/02092-9; 2022/02089-8), Prof. Edvaldo Sabadini (IQ-UNICAMP) and Prof. Camila Rezende (IQ-UNICAMP)

References:

[1] B. Mukherjee et al. **RSC Advances**, v. 6, n. 3, p. 1769-1781 (2016)

[2] L. Liu and F. Kong. **International Journal of Biological Macromolecules**, v. 139, p. 361-366 (2019)

[3] H.A. Barnes, J.F. Hutton, K. Walters. **An Introduction to Rheology**, 3ed., Elsevier Science Publishers B.V., Amsterdam (1993)

Influence of Polarization Time and Temperature on the Piezoelectric Properties of Complexe Geometry PZT Ceramics

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Piezoelectric materials have the capability of generating an electric potential in response to an applied mechanical stress (direct piezoelectric effect) or generating mechanical movement when subjected to an electric field (inverse piezoelectric effect) [1]. The piezoelectric ceramics are widely employed as sensors and actuators because of their idealelectromechanical properties. Their eletricmechanical properties make them suitable for use in devices such as hydrophones, static and vibration based energy harvesting devices,surgical tactile sensors, biomedical scaffolds and synthetic jet actuators [2]. Thepiezoelectric effect in polycrystalline ceramics is only obtained through the orientation of the ferroelectric domains by the temporary application of a strong electric field. This process is known as polarization [3]. In order to make the surfaces conductive, after sintering, the samples had their internal and external surfaces painted with silver paint andcured. In this article, the influence of time and temperature variation on the piezoelectric properties of PZT ceramics, for use in hydroacoustic transducers was evaluated. Currently,the following polarization parameters are used: electric field of 3kV/mm for 15 minutes atthe 100 ºC. Results indicated that the variation of time and temperature changed the piezoelectric properties, such as capacitance, electromechanical coupling and d33 parameter.

[1] J. Haoa, W. Li, J. Zhaia, H. Chen, Materials Science Engineering R 135 (2019) 1-57

[2] W. Du, J. Hoyt, N. Williams, K.A. Cook-Chennault. Journal of Manufacturing Processes 57 (2020) 48-60

[3] A. M.V.O. Ávila, M. A. P. Santos, F. L. de Magalhães, O. J. R. Afonso, T. O. A. Souza, G. A.C. Oliveira, X Encontro Técnico de Materiais e Química, Rio de Janeiro - 27 a 29 novembro 2019.

Influence of the high vacuum thermal annealing on the electrical and optical properties of ITO films grown by DC sputtering

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: Indium thin oxide (ITO) films are widely used as transparent conductors in various applications. In this work, tin-doped indium oxide films with different film thicknesses (305 nm, 692 nm and 1073 nm) were grown by the DC Sputtering technique. Subsequently, the as grown films were subjected to a thermal annealing treatment in a high vacuum condition. X-Ray Diffraction (XRD) data analysis revealed the formation of polycrystalline films with a bixbyite-type phase. It was determined a strong preferential orientation along the [222] direction for the thinner films. UV-Vis spectroscopy measurements indicated a transmittance value larger than 80% for the thinnest film, which decreases with the film thickness. In addition, room-temperature Hall effect measurements indicated that the increase of the electrons density leads to the transmittance decrease. It was determined high electron density values that surpass the maximum critical carrier density ($\sim 10^{19}\text{cm}^{-3}$); therefore, the electron-electron interactions become determinant and affect the optical band gap energy value. Electrical resistivity measurements carried out in the range 80-300K revealed the occurrence of a metal-insulator transition at $T\sim 113$ K. Further data analysis indicated the occurrence of quantum correlations associated with the electron-electron interactions, which show a consistent behavior determined from the optical measurements. It reveals that the electrical and optical properties of ITO films are strongly correlated, which is interesting for optoelectronic applications.

Investigating the Interaction between Quartz and Water using Molecular Dynamics Simulations with LAMMPS Software

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Quartz is a widely abundant mineral found in various types of rocks and present in all terrestrial regions [1]. It is known for its high thermal and chemical stability, as well as its hardness, making it a valuable material for a range of applications. Its interaction with water is also relevant due to its abundance in the Earth' s crust. Molecular dynamics is a branch of physics that studies the movements and interactions of molecules through modeling, simulation, and analysis. The LAMMPS software is a powerful tool for simulating atomic and molecular systems on a large scale using numerical integrators and a variety of interatomic potential models, such as Lennard-Jones potential, Morse potential, REAXFF, and the AIREBO potential. To investigate how fracture patterns affect wettability [2], thisstudy uses molecular dynamics simulations with LAMMPS. The system has periodic boundary conditions and is equilibrated with the NPT ensemble before strain is applied for100 ps to eliminate residual stresses. The system is also thermally equilibrated at ambienttemperature using the NVT ensemble for 100 ps. Strain is then applied with and withoutwater to evaluate mechanical properties, and other simulations are performed to assess thewettability. Overall, this study provides insights into the properties of quartz and its interaction with water, as well as the behavior of molecular systems using LAMMPS and theAIREBO and REAXFF potentials. The findings have implications for a range of fields, including materials science, geology, and physics.

Acknowledgements:
FAPDF, CNPq, CAPES, and UnB.

References:

[1] Klein, Cornelis. "mineral". Encyclopedia Britannica, 3 Feb. 2023, https://www.britannica.com/science/mineral-chemical-compound. Accessed 16 April 2023.
[2] Luz, A. B. D., Sampaio, J. A., França, S. C. A. (2010). Tratamento de minérios. CETEM/MCT.

Ion Exchange effects in Translucent Lithium MetasilicateGlass-Ceramics

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Lithium silicate glass-ceramics (GCs) are the most popular materials in the field of restorative dentistry. Translucency is an important feature that affects the esthetics of dental restorations. Transparent and translucent glass-ceramics are obtained when crystallizing nano-scale crystals specially if the refractive index of the crystals is similar to the vitreous matrix. However, the presence of nano-d crystals often leads to non-interlocked microstructures that render the material susceptible to catastrophic cracking under stress. Chemical strengthening by ion exchange is employed to enhance the mechanical properties without interfering in the optical properties. The improvement of mechanical properties occurs due to residual compressive stress induced on the sample surface by replacing smaller alkali ions in the glasses (e.g., Li and Na) by larger ones from a molten salt bath (K). In this work, we developed transparent glass-ceramics with crystallization of Li2SiO3 and Li2SiO5, and investigated the effects of the microstructure and ion exchange properties. Ion-exchange treatment was conducted at different times in two molten salts containing NaNO3 and KNO3 at 380 oC and 400 oC, respectively. The ion-exchanged specimens were analyzed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS) to investigate the microstructure and ion exchanged layer depth (DOL). The optical transmittance was evaluated using UV-VIS spectroscopy. Finally, samples were tested for flexural strength by the piston-on-three-ball method, Vickers hardness, and nano hardness. The high content of Li+ in the original glass allows for the formation of a great surface residual compressive stress. The flexural strength reached 800 ± 100 MPa (piston-on-three-ball) and Vickers Nanohardness of 11.6 ± 0.2 GPa (load of 200 mN), which are much higher than the glass-ceramic properties pre ion exchange 160 ± 50 MPa and 10.4 ± 0.2 GPa, respectively.

$La_{0.8}Sr_{0.2}Ti_{0.7}Ni_{0.3-x}Cu_xO_{3-\delta}$: Synthesis, characterization, and nanoparticles exsolution for SOFC anodes

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Climate emergency has become the century''s biggest concern due to the 1.5 ºC increase in the average temperature of the atmosphere, leading in the near future to a greater impact on natural events. In recent years, several efforts have been made to achieve net-zero emissions, based on the substitution of fossil fuels matrix by decarbonization. Solid Oxide Fuel Cell (SOFC) stands out as a viable alternative due to its high efficiency, fuel flexibility, and low-to-zero CO_2emissions. SOFC anodes are responsible for catalyzing the fuel reduction reaction. Among the used materials, perovskites are excellent candidates owing to their high mixed ionic-electronic conductivity (MIEC) and chemical and thermal capability. The present work seeks to obtain exsolved metal alloy nanoparticles anchored on perovskite structure [1] with the aim to address a more promising material, challenging and less contemplated in the literature. The nanoparticle catalysts were obtained by the interest metal solubilization into a perovskite host and exposed to a reducing H_2atmosphere. The $La_{0.8}Sr_{0.2}Ti_{0.7}Ni_{0.3-x}Cu_xO_{3-\delta}$(x = 0.1, 0.15, 0.2) powders were obtained by the Pechini synthesis route and calcined at 900 °C for 5 hours. To obtain decorated NiCu nanoparticles alloys, the powders were heat-treated under 3 vol % H_2/N_2atm at 900 ºC, 850 °C, 800 °C and 750 °C for10 hours. The samples were characterized by scanning electron microscope, X-ray diffraction, X-ray photoelectron spectroscopy, and electrochemical impedance spectroscopy. Rietveld refinement was used to determine structural parameters such as crystal symmetry, crystallite size, strain, and phase content before and after reduction treatment. The preliminary results revealed that the chosen synthesis route and the reduction treatment were successful in obtaining NiCu nanoparticles alloy.

Acknowledgments: CEM-UFABC, PRH49-ANP/UFABC, M2P-CINE-FAPES/Shell

References:

1-NEAGU, Dragos et al. Nature communications 6 (2015) 1.

Low-cost 3D printed solar panel dirt analyzer

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The installation of solar panels in external environments invariably leads to the accumulation of dirt on its glass surface, such as industrial or vehicular soot, salinity, sand, dust, organic materials, among others. As a result, efficiency reduction is observed, since the free light passage through the glass is compromised. In this work, CAD technology and 3D printing were used to develop a device to quantify the obstruction of the light passage in glass slides. Commercially available components were used, including Arduino, a luminous density sensor, a 1 W solar panel and an LED board to compose the prototype. The functionality of the device was demonstrated by quantifying the light passage and generated voltage after exposing glass slides samples to different external environments, for 30 days. The research results showed that it is possible to simulate the dirt accumulation on the solar panels glass surface, and based on this, maintenance cleaning can be schedule to avoid significant efficiency losses.

Acknowledgements:

The authors would like to acknowledge the support in financial resources and infrastructure by the companies ELEJOR S. A., ANEEL and Lactec, R D project 2945-1000/2017; CNPq process DT 1C, 30.8777/2020-4; CNPq Law 8010/90, DI 18/1041222-8, LI 18/1680554-2; and DI 19/0528208-9; LI 19/0641121-7; Lactec/IEP, UEPG/DEMA.

References:

AL-DOUSARI, A. et al., Energy, vol. 176, 184-194, 2019.

MICHELS, R. N. et al., Revista Agrogeoambiental, vol. 01, 126-129, 2009.

PARROTT, B. et al. Solar Energy, vol. 171, 526-533, 2018.

Luminescence modulation of carbon dots applied asouter layer in LEDs

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Light Emitting Diodes (LEDs) have revolutionized modern lighting due to their high luminous efficiency and less environmental impact. However, traditional LEDs are dependent on rare earth ions, which have high cost and nonrenewable resources [1]. In recent years, carbon dots (CDs) have been highlighted as new luminescent materials for making LEDs rare-earth-free. In this work, CDs were synthed from banana peel **(B-CDs)**and neutral red with citric acid **(Nr-CDs)**for luminescence modulation to generate a fluorescent film and apply as an outer layer in LEDs to improve the electrical properties. In this sense, **B-CDs**and **Nr-CDs**were embedded in the polymer (PVA; poly (vinyl alcohol)) to obtain the film **(BNr-CDs-F)**. All CDs exhibited absorption bands related to π → π* and n → π* characteristic of the graphitic structure and surface functional groups, respectively.[2]**B-CDs**showed excitation-wavelength-dependent emission with maximum intensity at 454 nm (λ_{exc}= 360 nm). On the other hand, **Nr-CDs**displayed intriguing dual-emission behavior at 450 and 600 nm. In addition, **Nr-CDs**and emission modulation of both CDs (**B-CDs**and**Nr-CDs**) exhibited fluorescence close to the CIE chromaticity coordinates (x,y) for pure white light emission (0.33, 0.33). **BNr-CDs-F**film was applied as an outer layer on the UV LED (λ_{exc}= 200 nm, 50 cd.m^{-2}). Except for the B-CDs film, the use of the **Nr-CDs**and **BNr-CDs-F**films amplified the efficiency of the commercial LED around 2 (109 cd.m^{-2}) and 7 times (374 cd.m^{-2}), respectively. Furthermore, different white color variations were obtained by changing the power of the LED. Finally, the results obtained indicated that the outer layer produced by modulating the fluorescence of the CDs was efficient to increase the efficiency of commercial LED.

Acknowledgments: CNPQ, CAPES, UFAL, FAPEAL and CTI/MCTI.

References

[1] Zhao, B., Zhan' ao, T. Advanced Science, vol 8, 2001977 (2021)
[2] Shalmali Hui, J Nanopart Res, vol 25 (2023)

Luminescence properties of Y2O3 doped with Eu and Tb produced by an eco-friendly method

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Materials produced via eco-friendly routes has been highlighted in recent years [1]. In this work, the modified sol-gel route using organic matter from river water was used to produce nanomaterials. The material of interest is yttrium oxide (Y2O3) undoped, doped, and co-doped with europium and terbium ions. Y2O3 is considered a great host matrix for rare-earth ions to improve their luminescent properties, consequently, it can be used in the manufacture of lasers, sensors, solar cells, among others. The samples were calcined at a fixed temperature of 1000 ºC, and the parameters investigated in the initial part of the work were the doping concentration and the reproducibility of the synthesis by using river water from different origins. The explored concentrations were: 1%, 2% and 5%. Powder XRD measurements, showed that single crystalline phase were obtained for all samples regardless of the origin of the river water, which indicates good reproducibility of the Y2O3. Furthermore, when estimating the average crystallite of the sample produced with distilled water and river water, 26 nm and 31 nm were obtained, respectively. The diffraction patterns of the doped samples did not show secondary peaks, which implies that the doping was successfully performed. Photoluminescence measurements were performed, and the emission spectrum presents the characteristic peaks of Eu and Tb. For the excitation spectrum, several bands from 170 up to 466 nm were observed, making this material a potential candidate for many applications, such as solar cells and luminescence thermometers.

Acknowledgments: This work has been supported by the Brazilian funding agencies CNPq, CAPES, FINEP and FAPITEC. This research used facilities of the CMNano/UFS under proposal 080/2023.

Reference:

[1]. GOMES, M.A. et al. "Temperature-sensitive luminescence of Y2O3:Nd3+ nanocrystals produced by an eco-friendly route", Optical Materials, v. 89, p. 536-542, 2019.

Mechanical performance of cementitious composites reinforced with nylon microfibers produced by solution blow spinning

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Reinforcing fibers in concrete structures have been successfully applied due to their superior mechanical properties and anchoring capacity. In the case of synthetic polymers [1], nylon 66 (polyamide 66, PA66) is a multifunctional synthetic thermoplastic polymer, being tested today for use as reinforcement in cementitious composite engineering with good cost-benefit results. In this field, the fibers can act on the cracked surface as a bridgeeffect, providing residual tensile strength [2]. Furthermore, with the use of nanostructuredfibers, new properties emerge due to the high surface area, the elongated length and the high degree of entanglement. For the rapid fabrication of nylon 66 fiber materials, solutionblow spinning (SBS) technique [3] was used, which has a high efficiency and low cost. Thefibers were characterized by Scanning Electron Microscopy (SEM) and Fourier TransformInfrared Spectroscopy (FTIR) and dispersed in a cementitious matrix. SEM results showedfiber morphology obtained and FTIR showed strong hydrogen bonding interaction. The compressive strength of concrete was higher in samples with micrometric nylon fibers. Finally, tests indicate that the use of SBS enabled the production of nylon 66 fibers quickly and efficiently, which can be used as suitable choices to produce cementitious composites with acceptable performance and low prices for usual applications in the construction industry.

Acknowledgements:

Authors are grateful to CNPQ/Brazil (Process 408905/2022-0 and 308353/2022-6), FACEPE (APQ-1251-3.03/22) and CAPES/Brazil (finance code 001) for partially funding this work and scholarship.

References:

[1]Z. Yang, C. Shen, Y. Zou, D. Wu, H. Zhang, K. Chen, Foods 10 (2021) 2339.

[2]C. Lin, T. Kanstad, S. Jacobsen, G. Ji, Constr. Build. Mater. 378 (2023) 131169.

[3]E.S. Medeiros, G.M. Glenn, A.P. Klamczynski, W.J. Orts, L.H.C. Mattoso, J. Appl. Polym. Sci. 113 (2009) 2322-2330.

Mechanochemical activation for obtaining hierarchical ZSM-5 zeolite using dual templates

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Hierarchical zeolites are a class of materials that exhibit unique properties due to their porous nature, which allows for the selective adsorption and catalysis of molecules [1]. The ZSM-5 zeolite is one of the most widely studied due to its high acidity and shape-selective catalysis [2]. The goal of this work is to develop hierarchical zeolites from mechanical activation of NH4ZSM-5 (Si/Al=23) using two templates: tetrapropylammonium bromide (TPA) and cethyltrimethylammonium (CTMA) in the absence of solvents. The reactants were mixed in a ball mill (IKA instruments, Ultra Turrax Tube Drive UTTD), and milled varying the time from 0 to 20 min. The calcined materials at 550 oC were analyzed by X-ray diffraction (XRD), infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The XRD showed well defined pattern for MFI structure, indicating that there was no amorphization under the mechanical treatment. This was confirmed by the SEM and FTIR. The ZSM-5 activated by 10 and 15 min exhibit a small increase of the crystallite , calculated by the Scherrer equation, whereas for 5 min was observed a loss in crystallite , suggesting that the treatment at this time is enough to break the siloxane bond (Si-O) and create space for the TPA and CTMA fill the pores generated through the ball milling. At times higher than5 minutes, is proposed a breaking of Si-O-Si bonds followed by recrystallization and possible increase of the of crystallite over the time.

Acknowledgements: PRH/ANP 37.1 for the resources and reagents.

References:

[1] S. Mardiana, N J. Azhari, T. Ilmi and G.T.M. Kadja., Fuel 309, 122119 (2022).
[2] D.N. Rainer am R.S. Morris., Dalton Trans. 50, 8995 (2021).

Methane derived carbon nanotubes by chemical vapor deposition

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Carbon structures are widely studied mainly due to their electronic properties, which allow their application in supercapacitors, energy harvesting, sensors, and other electronic devices. The possibility of preparing these materials from methane gas makes the idea even more interesting. Furthermore, carbon structures can be formed on different substrates, such ceramic fiber paper (CFP). This substrate is flexible, chemically stable and withstands high temperatures (~1,200 °C) [1]. Thus, in this work, we synthed carbon structures from methane on CFP by the chemical vapor deposition (CVD) method. The CVD was performed in a sample of CuO impregnated CFP. To produce the CFP-CuO sample, pieces of CFP were quickly immersed in an alcoholic solution of copper salt and dried in an oven. After that, the sample was calcined to form the CuO phase. Then, a tubular oven with nitrogen and methane gas flow was used to perform the CVD method [2]. The CFP-CuO sample was inserted into the tubular oven, the nitrogen gas flow was used to clean the atmosphere inside the tube. The oven was heated to and the methane gas flow was opened for 10 min. After that, the methane gas flow and the heating were stopped. The nitrogen gas was used to clean the tubular oven again and the sample was recovered at room temperature. The samples were characterized by X-ray diffractometry (XRD) and scanning electron microscopy (SEM). The XRD confirmed the formation of CuO and carbon on the samples. The SEM images showed that carbon nanotubes grew on the CFP-CuO.

Acknowledgements:
The authors acknowledge São Paulo Research Foundation (FAPESP) (grants 2018/01258-5, 2020/05233-7).

References:
[1] Schawaller, Dirk et al 2012. Macromolecular Materials and Engineering, v. 297, n. 6, p. 502-522.
[2] LE, Huu Doan et al 2013. Advances in Natural Sciences: Nanoscience and Nanotechnology, v. 4, n. 3, p. 035012.

Microscopic electrical conduction in carbon-black materials

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The electrical properties of carbon-blacks materials have been intensively studied. This powder material is largely used in composites, pigments, coating applicatins, UV shielding, and etc. Carbon-blacks are made of nearly spherical carbon nanoparticles fused into chain-like structures called aggregates. Therefore, the electron conduction should depend on the capacitance between neighbor aggregates. In this work we have modeled the aggregates with defined fractal dimension, the inter-aggregate capacitance and the space distribution of the path channels used by an conduction charge. Our results will be useful to better explain the electron conductivity in carbon-black samples.

Microstructural evolution of castable high-alumina ceramics bonded with anionic colloidal silica

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Ceramic materials based on mullite (Al6Si2013) are used in high-temperature-based industrial processes due to resistance to corrosion and refractoriness. In this work, samples of alumina-mullite were produced from a dispersion of anionic colloidal silica and calcined alumina particles processed by the direct casting method and treated isothermally in the 120-1500°C temperature range. Samples were submitted to non-destructive tests for evaluating the physical, chemical and microstructural properties. The drying behavior established parameters that ensure a sufficient level of mechanical strength to hand green samples. After drying, CS-bonded samples presented higher elastic modulus than the calcium aluminate cement and hydratable alumina-bonded ones, tested as references under the same conditions. Increasing isothermal treatments' temperatures, the stiffness modulushas increased, differently from the behavior shown by the other binders. Crystalline phasesrelated to cristobalite and mullite formation were detected above 900°C. Regarding the microstructures, the dry samples showed superficial microcracks. Although they grew with the increase in the firing temperature, they did not compromise the mechanical properties of the parts.

Acknowledgment: FAPESP (2010/19274-5), CNPq (147526/2021-3), CAPES (PNPD 88887.648527/2021-00), ALMATIS (Brazil and Germany), Nouryon SA (Brazil).

References:

[1] STUDART, A. R.; GONZENBACH, U. T.; TERVOORT, E.; GAUCKLER, L. J. Processing routes to macroporous ceramics: A review. Journal of the American Ceramic Society, v. 89, n. 6, p. 1771-1789, 2006;

[2] SALOMÃO, R.; ARRUDA, C. C.; SOUZA, A. D.V.; FERNANDES, L.; Novel insights into MgO hydroxylation: Effects of testing temperature, samples′ volume and solid load; Ceramics International, v. 40, 14809 - 14815, 2014;

[3] L. L. Sousa et al, Development and characterization of porous moldable refractory structures of the alumina-mullite-quartz system, C. I., vol. 43, n. 1, 2017.

Modeling emissions by rate equations and evaluating relative thermal sensitivity of a ytterbium-erbium codoped nanoparticle system

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Temperature is a crucial parameter across a wide range of phenomena and fields, including physics, biology, medicine, and more [1]. Specifically, in nanomedicine, temperature plays a significant role in both diagnostic and therapeutic applications, both in vitro and in vivo [2]. For instance, real-time temperature monitoring is critical in preventing healthy tissue overheating during photothermal therapy [3]. Motivated by these and other factors, our objective was to use rate equations to model the emissions of a system (LaF3 nanoparticle) codoped with 18 wt% of Ytterbium and 2 wt% of Erbium ions, both trivalent. The simulations were performed using the Octave software. As a result, we found that the analyzed emissions showed a behavior in excellent agreement with those obtained in experiments. Furthermore, we evaluated the relative thermal sensitivity of the system by analyzing the fluorescence intensity ratio (FIR) between the emissions at 520 and 540 nm. A maximum relative thermal sensitivity of 0.14%/K at 425 K was obtained, which is consistent with values reported in the literature.

Acknowledgments

We acknowledge the financial support from Brazilian Agencies: FINEP (Financiadora de Estudos e Projetos) through the grants INFRAPESQ-11 and INFRAPESQ-12; CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnologico) through the grants: Projeto Universal Nr. 431736/2018-9 (C. Jacinto); CNPq by means of the PIBIC/2022-2023 scholarship of the R. C. Silva.

References

[1] D. Jaque and F. Vetrone, Nanoscale 4(15), 4301-4326 (2012).

[2] D. Jaque, B. del Rosal, E. M. Rodríguez, L. M. Maestro, P. Haro-González, and J. G. Solé, Nanomedicine 9(7), (2014).

[3] D. Zhi, T. Yang, J. O’ Hagan, S. Zhang, and R. F. Donnelly, Journal of Controlled Release 325, 52-71 (2020).

Morphology tailoring of oxide particles by gas pressure hydrothermal synthesis

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The properties of oxide particles are directly related to their morphologies [1]. This work presents a new adaptation of the hydrothermal synthesis method, which can promote morphological changes in the synthed materials (ZnO and Mordenite) by the injection of pressurized inert gas [2]. ZnO particles were synthed at 120°C for 20 h and pressures of 1.4bar (autogenous) and 50 bar (argon), while mordenite was processed at 170°C for 20 h and pressures of 6.5 bar (autogenous) and 25 bar (argon). SEM and XRD analyses showed that both materials underwent significant changes in their morphologies, different from those achieved under normal synthesis conditions (autogenous pressure), while their crystalline aspects remained unchanged.

Acknowledgements:

To the Human Resources Program of the National Agency of Petroleum, Natural Gas and Biofuels - PRH-ANP, for the financial support supported with resources from the investmentof oil companies qualified in the RD I Clause of ANP Resolution nº 50/2015.

References:

[1] M. Nakayama, K. Nishii, K. Watanabe, N. Tanibata, H. Takeda, T. Itoh, T. Asaka, Sci. Technol. Adv. Mater. Methods. 1, 24-33 (2021).

[2] A.M. Mendes, A.E. Martinelli, Mater. Lett. 256, 126597 (2019).

Nanoreactor approach in the preparation of carbon nanoparticles

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The conventional synthesis of carbon nanoparticles (CNPs) is low-temperature pyrolysis of individual organic compounds under solvothermal or microwave-assisted treatment. Due to different carbon sources and synthetic conditions, CNPs commonly have different sizes, surface chemical composition, and photoluminescent properties. Thus, these approaches are unable to ensure the reproducibility of the synthesis. The primary strategy to improve synthetic conditions is spatial isolation of the carbon sources in nanoreactors. The confined space of nanoreactors can increase the probability of reaction collision and the mass transfer of reactive chemical molecules, thereby significantly improving the efficiency of chemical reactions. The current presentation will discuss various methods used for spatial isolation of the precursors in CNDs synthesis. The preparation of CNDs using vesicles and micelles is one of the nanoreactor strategies. It was demonstrated that sugar carbonization inside reverse micelles built from bis(2-ethylhexyl) sulfosuccinate resulted in fluorescent CNDs with 1.8–4.0 nm in size. Fatty acids were used as amphiphilic nanoreactors obtainingin situ upon irradiation (Xe lamp 500 W 4–12 h). When nonanoic acid was used as a carbonsource, CNDs with particle size 1.6 nm were obtained despite the vehicle''s large size (100nm). Another example of the nanoreactor approach is the utilization of mesoporous metal–organic frameworks (MOFs). Depending on MOFs'' cave size, CNDs with sizes of 1.5, 2.0, and 3.3 nm can be obtained from glucose. Several attempts to use molecular sieves and other microporous materials as templates for CNDs synthesis were also described. In most cases, there were essential contradictions between the size of the pores of the template and the size of the carbon particles received. The application of mesoporous materials to confine the growth and shape of carbon nanoparticles and their aggregation is rapidly developed in recent years.

Nanostructures of CuO and CuO/ZnO in the Productionof Biodiesel from Residual Cooking Oil

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Biodiesel is a biofuel that could replace fossil diesel. It is extracted from biomass which is considered a renewable source of energy, therefore it is a clean fuel. Biodiesel production is practiced at a global level, and Brazil has the advantage of its great energy potential for this purpose. Oilseeds, animal fats and residual oils from frying are used as raw materials. This potential also extends to the research and development of catalysts that contribute to improve yields and increase the speed of chemical reactions in the production of biodiesel. Among these, esterification is used to obtain alkyl esters, better known as biodiesel. Thus, this work aimed to initially use 100% CuO and 80% CuO catalysts with the addition of 20% ZnO as test parameters to produce biodiesel from residual cooking oil. The catalysts were synthed through the combustion reaction and were characterized by X-ray diffraction (XRD), infrared spectroscopy (FTIR) and thermogravimetric analysis (TG). After the characterization of the catalysts, the esterification reaction of the residual frying oil was carried out, aiming at its conversion into biodiesel. Finally, the efficiency of the catalysts was verified by the analysis of gas chromatography, which obtained a result of conversion of residual oil into biodiesel of 17.95% for CuO and 80.4% for CuO80/ZnO20. The results of the structural and thermal characterizations revealed that the metallic oxides used as catalysts have suitable properties for application in the production of biodiesel, which achieves greater sustainability as a result of the recovery through the use of residual biomass offered in this work.

Acknowledgements:

The authors are grateful to PNPD/FAPESQ, beneficiary program Renewable Energy, for the financial support.

References: [1] J. Dantas, E. Leal, D. R. Cornejo, R. H. G. A. Kiminami, A. C. F. M. Costa. Arabian Journal of Chemistry, v. 13, p. 3026-3042 (2020).

Ni nanostructures formed by pH-dependent corrosion-redeposition of thin nickel films using glycine solutions

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Properties such as resistance to high temperatures, oxidation and corrosion make nickel (Ni) an interesting material for many applications, which include Ni nanostructures. For example, applications of Ni nanowires range from optical devices and solar cells to sensors and biotechnology. Several methods have been used so far for the synthesis of Ni nanostructures, such as hydrothermal, laser ablation and chemical vapor deposition [1,2]. In this work, we explore a different method for the synthesis of Ni nanostructures, based on local corrosion of thin Ni films prepared by physical vapor deposition. We demonstrate and explore the slow Ni corrosion produced by solutions containing water, glycine and calcium chloride, with different pH values. After a relatively long (48h) contact time between Ni film and solution, we observe isotropic corrosion patterns with approximately circular shapes. Within the surface patterns, nanostructures are formed; usually a nanowire-like structure projects vertically from the center of most patter for the larger patterns, very thin planar nanowires, with a fractal aspect, originate from the structure at the center. Energy Dispersive Spectroscopy measurements carried out with Scanning Electron Microscopy shows that the central nanostructure is composed of Ni, while the composition of the thinner, planar nanowires can not be resolved. Our results point to a process of corrosion at local defects of the film, and redeposition due to the radial gradient of Ni dissolved in the solution, which is kept static during the whole time. Further studies can determine if this process can be made controllable as a new synthesis process of Ni nanostructures.

Acknowledgements:This work was financially supported by the Brazilian funding agencies CNPq and Fapesp (grant number 2019/07616-3).

References:

[1] Y.Y. Kong, S. C. Pang, S. F. Chin, Materials Letters 142 (2015), 1-3

[2] D.P. Wang, et al. Materials Chemistry and Physics 113 (2009), 227-232

Nitrogen-Doped Graphene Mechanical Properties

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Graphene is a two-dimensional material composed solely of carbon atoms bonded in a hexagonal structure. Its unique properties, such as high electrical conductivity and rigidity, make it a material of great interest in scientific research. Nevertheless, some applications still require tailor specific properties. One interesting approach to this end is doping graphene with atoms of other substances, such as nitrogen, which can further improve its properties, making it even more versatile in terms of applications. However, the nitrogen doping process can weaken the graphene sheet since carbon-nitrogen bonds are weaker than carbon-carbon bonds. Also, excessive doping can lead to material rupture, rendering it unusable. In this context, this work applies molecular dynamics simulations to investigate the effects of nitrogen-doped graphene on mechanical and topological properties with different doping percentages (1 to 4%), temperatures (100 to 900 K) and defects (0.05 to 0.4%). The graphene sheet was doped with nitrogen by replacing randomly carbon atoms with nitrogen atoms, while defects were inserted by randomly removing carbon atoms using a 17nm x 17nm structure containing 11200 particles. The results showed that the doping of graphene with nitrogen decreases the material's strength by approximately 14% compared to pristine configuration. However, it was not observed a significant difference in mechanical resistance with the doping level. As expected, the increase in temperature and defects also decreases the doped graphene's strength, reaching a maximum reduction of around 28% and 32%, respectively. Finally, all studied configurations remained stable during the mechanical testing, demonstrating that they might be suitable for further experimental analysis.

Acknowledgments:
The authors are thankful for the financial support of the Brazilian agencies FAPERJ and CNPq.

Novel hierarchical materials with transition metal cations for immobilizing laccases to remediate textile industry pollution

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The synthesis of zeolites materials containing metals such as Al, Y, V, Sn, Nd, Nb and Zr in octahedral;pentahedral;tetrahedral coordination (OPT' s) composing the crystal lattice of the material, received increasing attention over the years [1-2]. In this work, different hierarchical zeolites containiung transition metal based materials are reported. The syntheses of these materials were adapted from earlier reports described elsewhere [3]. The use of tetra propyl ammonium bromide (TPABr) known as a structure-directing agent (SDA); Bismuth (III) nitrate pentahydrate (Bi(NO3)3.5H2O), V2(SO4)3 and $SnCl_2$. $2H_2O$ as metal source Ludox HS-30 colloidal solution as a source of silica (SiO2) and pellets of sodium hydroxide (NaOH) as a mineralizing agent, were utilized to produce the material. The materials were silanized utilizing APTES and APTMS, and functionalized afterwards with glutaraldehyde to immobilize the laccases from Trametes villosa and Pycnoporous sp. To removal of textile dyes by adsorption and biodegration [4]. The results obtained suggested that the application of a new and hierarchical material containing bismuth in its framework as a physical support was successful, being possible to retain the enzyme in this material and to apply as removal agent of the dyes in aqueous solution. Bioinformatics studies was conducted to help us understand the mechanism of how laccases interact with the hierarchically structured supports, allowing to make an optimization of the immobilization process and the immobilized enzyme activity.

Acknowledgements:
We would like to thank FAPESP, CAPES and CNPq for supported this work.

References:

[1] ROCHA, J. and Lin, Z., RIMG 57 (1): 173-201 (2005).
[2] ROCHA, J. and ANDERSON, M. W, EurJIC 2000 (5) 801-818 (2000).
[3] ZANARDI, S. and CARATI, A. Microporous and Mesoporous Materials 97 (1-3): 34-41 (2006).
[4] MODKOVSKI, T. and MACIEL, G. Water Air Soil Pollut 232:512 (2021).

Obtaining self-aggregating systems from sodium cholate/lecithin

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The study of systems formed by surfactants has been increasing in recent years, the great interest in these types of structures is due to their broad biochemical and biomedical potential. The mixture of surfactants can lead to the formation of different aggregates, such as wormlike micelles, reverse micelles, liposomes and vesicles [1]. The bile salts, such as sodium cholate, are natural amphiphilic surfactants, stored in the gallbladder, and act to emulsify and lipids to facilitate digestion [2]. Lecithin is a phospholipid, a naturally occurring zwitterionic surfactant, found in egg yolks and soybeans, and can form reverse wormlike micelles when incorporated into other molecules [3]. Sodium cholate/lecithin systems were prepared in different ratios (0.2; 0.4 and 0.8 (m/m)). They were also prepared with two solutes: polyethylene oxide or propylene glycol, to verify changes in the arrangement and dimension of aggregates. In this sense, the and shape of the aggregates were determined by Dynamic light scattering (DLS), small angle X-ray scattering (SAXS) and rheology studies were performed to determine the parameters such as relaxation time, modulus at the plateau, elastic modulus, viscous modulus and frequency.

Acknowledgments:
FAPESP (22/02092-9; 22/02089-8), CAPES (88887.713913/2022; 0088887.713911/2022-00) and Professor Dr. Edvaldo Sabadini (UNICAMP).

References:

[1] Lee, Y. K. and Lee, H. Y. **Colloids and Surfaces A.**v. 538, p. 661 - 667, 2018.
[2] Cautela J. et al. **Colloids and Surfaces A**, v. 532, p. 411-419, 2017.
[3] Tung, S. H. et al. **Journal of the American Chemical Society**, v. 128, n. 17, p. 5751-5756, 2006.

Optical properties of self-assembled InAs quantum dots based P–I–N structures grown on GaAs and Si substrates by Molecular Beam Epitaxy

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Extensive work on InAs quantum dots grown on GaAs substrates has been reported in the literature. However, research in the use of different substrate materials such as silicon to achieve an ideal and full integration of photonic and electronic systems is still a challenge. In this work we have investigated the effect of the substrate material (Si and GaAs) and strain reducing layer on the optical properties of InAs quantum dots for possible applications in laser devices grown by Molecular Beam Epitaxy. Two InAs quantum dots structures with similar active regions grown on GaAs and Si substrates using strain reducing layer consisting of InAs QDs/6 nm $In_{0.15}Ga_{0.85}As$ have been investigated. Atomic Force Microscopy, Transmission Electron Microscopy, and photoluminescence have been used for the characterization of the samples. We have observed a red shift of the InAs QD photoluminescence peak energy for the sample grown on Si substrate as compared to the sample grown on GaAs substrate, which was associated with residual biaxial strain from the Si/GaAs heterointerface. The red-shift of the photoluminescence peak energy is accompanied by a broadening of the photoluminescence spectrum from ~31 meV to a value of ~46 meV. This broadening is attributed to the quantum dots inhomogeneity increase for samples grown on Si substrate. This result open new insights for the controlling the emission of InAs quantum dots for photonic device integration using Si substrates.

The work has been supported by Fundação de Amparo a Pesquisa do Estado de São Paulo (FAPESP) (grants no. 19/23488-5 and 19/07442-5), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq)

Physical and microstructural characterization of recycled aggregate obtained from geopolymer concrete

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Geopolymers are inorganic materials composed of a source of aluminosilicates together with an alkaline solution, and have high adhesion due to their rheology and structure when applied as a matrix in concrete and mortar [1], [2]. The recycling of concrete as an aggregate is the subject of studies due to the incorrect disposal of the second most produced artificial material in the world [3]. In the present study, the density, water absorption, and microstructure of aggregates produced from geopolymeric concrete were evaluated. The geopolymer concrete was fragmented, sieved and segregated to obtain the gravel granulometry 0, above 4.75 mm. After this process, the aggregates were submitted to physical tests of water absorption and density, and analyzed by optical microscopy. The aggregate showed a high water absorption, about 17%, and the density was about 5,165 kg/m3, and the microstructure showed low adhesion of the aggregate already present in the aggregate coming from the concrete, which causes a low resistance.

Acknowledgmet:

CAPES, CNPQ, PROPESP/UFPA, and the laboratories that comprise the composite materials research group of UFPA.

References:

[1] Tam, Vivian WY, Mahfooz Soomro, and Ana Catarina Jorge Evangelista. A review of recycled aggregate in concrete applications (2000-2017). Construction and Building materials 172 (2018): 272-292.

[2] Davidovits, J. (2002, October). years of successes and failures in geopolymer applications. Market trends and potential breakthroughs. In Geopolymer 2002 conference (Vol. 28, p. 29). Saint-Quentin, France; Melbourne, Australia: Geopolymer Institute.

[3] Thomas, B. S., Yang, J., Bahurudeen, A., Chinnu, S. N., Abdalla, J. A., Hawileh, R. A., ... Hamada, H. M. (2022). Geopolymer concrete incorporating recycled aggregates: A comprehensive review. Cleaner Materials, 100056.

Piauí Kaolinite: Characterization Studies forApplications In Environmental Remediation

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Kaolinite, a naturally occurring mineral clay with chemical formula $Al_2[Si_2O_5](OH)_4$, with good availability and low cost, is widely used in industry as an additive for polymeric compounds, filler and coating for ceramic materials [1]. Kaolinite shows high porosity, high adsorption capacity and plenty of hydroxyl groups capable of participating in many chemical reactions, as well as ionic exchange processes. Such properties qualify this mineral clay as a good adsorbent for removing pollutants (dyes, for example) from wastewater [2]. The lack of studies on the application of kaolinite (available in the Picos microregion, specifically in the municipality of Oeiras, in the state of Piauí-Brazil) to studies of environmental remediation,as adsorbent, became an attractive and a relevant issue. So, it is important to understandthe physicochemical properties of kaolinite from that region. Using characterization techniques, such as XRD, FRX, FTIR, SEM, BET, TG and Zeta potential, one can evaluate the chemical and physical structures as well as the morphology and compare them with the data from literature. Considering the results in initial adsorption tests of methylene blue (as a model dye) on powders of kaolinite from Piauí-Brazil, it can be concluded that this materialis very promising.

Acknowledgements: CAPES, CNPQ, UFPI, IFCE, LIMAV

References:

[1] CHENG, H.; ZHOU, Y.; LIU, Q. Kaolinite Nanomaterials: Preparation, Properties and Functional Applications. Nanomaterials from Clay Minerals: A New Approach to Green Functional Materials, p. 285-334, 2019.

[2] JAWAD, A. H.; ABDULHAMEED, A. S. Mesoporous Iraqi red kaolin clay as an efficient adsorbent for methylene blue dye: Adsorption kinetic, isotherm and mechanism study. Surfaces and Interface, v.18, p. 100422, 2020.

POST-CONSUMER PET/HDPE RECYCLING BY EXTRUSION AND INJECTION

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Polymers such as high-density polyethylene (HDPE) and polyethylene terephthalate (PET) are widely used, generating large amounts of post-consumer waste($_{PC}$)[1], requiring conscious innovations in raw materials, products and technologies [2] .The objective of this work was to produce HDPE/PET$_{PC}$polymeric blends, for the company COGUMELO, which currently only recycles HDPE$_{PC}$. Were extruded 6 blends$_{PC}$: A1.1-60HDPE/40PETCR, A1.2-60HDPE/40PETIG, A2.1-70HDPE/30PETCR, A2.2-70HDPE/30PETIG, M1.1-50HDPE/50PETCR, M1.2-50HDPE/50PETIG and PEAD$_{PC}$(range 140-240 ºC and 140-150 RPM) for the production of specimens in a heated hydraulic press (thermal analysis: HDT and VICAT) and injection in a closed mold (COGUMELO mechanical test - Deflection: 900mm clearance). Results obtained for HDT(℃)-HDPEPC:66.6, A1.1:75.6, A1.2:79.8, A2.1:73.3, A2.2:75.4, M1.1:77.0, M1.2:87.0;VICAT(℃)-HDPEPC:110.5, A1.1:116.1, A1.2:119.7, A2.1:119.1, A2.2:117.8, M1.1:124.6, M1.2:123.6 and Deflection (mm)-HDPE$_{PC}$: 3.94, A1.1:3.03, A1.2:2.84, A2.1:3.09, A2.2:3.44, M1.1:2.31, M1.2:2.05. The incorporation of PET$_{PC}$in the blends promoted an increase in the values of thermal and mechanical resistance, compared to the values of the HDPE$_{PC}$matrix, provided by the benzene group responsible for the stiffness of the PET chain, giving the material greater thermal and mechanical resistance in the solid state[3].Acknowledgements: FAPERJ, COGUMELO, UERJ-ZO.References:[1] ITO,E.N.;BALABAN, R.C.Comportamento Mecânico de compósitos a base de blenda de PET reciclado/PEAD reforçados com fibras de bananeira roxa.1ªed.São Carlos:Associação Brasileira de Polímeros Regional Nordeste, 2014, v.1, p.45-48.[2]ABIPLAST.Associação Brasileira da Indústria do Plástico, 2019. Disponível em: .Acesso em: abril de 2023.[3]ROMAO,W.;SPINACÉ,M.A.S.; PAOLI,M.A. Poli(tereftalato de etileno),PET:uma revisão sobre os processos de síntese, mecanismos de degradação e sua reciclagem. Polímeros, v.19, p.121-132, 2009.

Production of Scheelite (CaWO4) sheets by aqueousTape Casting for dielectric applications

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Scheelite, or calcium tungstate ($CaWO_4$), is a mineral which represents great economic importance for responding to one of the largest quantities of tungsten production, being widely found in the Seridó region of Rio Grande do Norte, Brazil [1]. In recent years, Tungsten-based materials have gained attention of the industry due to their unique physical-chemical properties and their excellent electrochemical performance, being often used as components of energy storage and saving devices [2]. Parallel, the tape casting method is a well-established method to produce thin and flexible sheets, often applied in electronic industry [3]. Therefore, the aim of this research is to produce Scheelite sheets by aqueous tape casting and to evaluate the dielectric properties and applications. The Scheelite powder was obtained in Brejuí Mine, and characterized by X-ray fluorescence and diffraction analysis. The rheologic behavior of the suspension was measured, in addition to the dielectric properties of the sheets. SEM and EDS analysis were performed to evaluate the characteristics and composition of the surface of the sheets. The obtained results satisfied the expectations for the desired applications.

Acknowledgments: This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) - Finance Code 001.

References:

[1] NAVARRO, Gulhermo Rafael B. et al. 2017. Livro de referência de Minerais Comuns e Economicamente Relevantes: WOLFRAMATOS. Museu de Minerais, Minérios e Rochas Prof. Dr. Heinz Ebert.

[2] SHINDE, P. A.; JUN, S. C. Review on Recent Progress in the Development of Tungsten Oxide Based Electrodes for Electrochemical Energy Storage. ChemSusChem, v. 13, n. 1, p. 11-38, 9 jan. 2020.

[3] JABBARI, M. et al. Ceramic tape casting: A review of current methods and trends with emphasis on rheological behaviour and flow analysis. Materials Science and Engineering: B, v. 212, p. 39-61, 2016.

Production of silicon carbide (SiC) using rice husk and microwave radiation

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The production of silicon carbide is known for using a high temperature process, known as the Acheson process. An alternative route that has been studied is a synthesis using microwave radiation, where the mechanical waves generated by the frequency of 2.45 GHz are capable of propagating heat through phonons. Studies based on microwave-assisted synthesis are promising and more economical than the previous one. [1]. The rice husk is a product of the production of rice and is abundant in all of the rice producing countries and is generally treated as a waste. In its composition, there are 50% of organic carbon and 15% to 20% of SiO_2[2]. After the carbonization process of the rice husk, the ashes generated contain around 90% of SiO_2[3]. The aim of this project is to produce SiC from rice husk provided by a rice mill in the south of Brazil. The raw material was milled and characterized using X-ray fluorescence spectroscopy (XRF), Scanning electron microscopy (SEM) and Thermogravimetric analysis (TGA). The characterized rice husk was carbonized after the milling processes in a furnace at temperature of 600ºC for 3h and the material obtained was 41,6% of SiO_2. This same material was submitted in a loss of ignition analysis at a temperature of 1.000ºC for 5h. This material had 55% of its mass of loss in ignition material and the ashes remaining were about 85% of SiO_2. The results confirmed the percentage of SiO_2 obtained in previous studies.

Acknowledgements: CAPES

References:

[1] Li, J., Shirai, T., Fuji, M. (2013). Rapid carbothermal synthesis of nanostructured silicon carbide particles and whiskers from rice husk by microwave heating method. Advanced Powder Technology, 24(5), 838-843.

[2] Singh, B. (2018). Rice husk ash. Waste and Supplementary Cementitious Materials in Concrete, 417-460.

[3] PEREIRA, M. L. G. et al. (2020) Synthesis of silicon carbide from rice husk. Cerâmica, v. 66, p. 256-261.

Properties of Piauí attapulgite for adsorption and desorption of phosphorus from sludge

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The sustainable development of materials for environmental remediation with applications in agriculture has aroused interest. For example, the recovery of phosphorus (P) from sludge for use as fertilizer. Attapulgite, a hydrated magnesium clay mineral abundant in thestate of Piauí, is effective as a P adsorbent material, by several mechanisms, due to its chemical composition and morphology [1]. Initially, the attapulgite was characterized by XRD, where peaks characteristic of this mineral was observed in the region of 8.46° and 20.21°, by FRX highlighting the percentages of 38.51% of SiO_2, 9.15% of MgO, and 15.49% of CaO and by SEM, where a fibrous morphology is observed. In addition, the methodologyfor recovering the P in the sludge is imperative, focused only on the removal andimmobilization of this nutrient in its various forms. In this context, the hydrothermal treatment proved to be effective in improving the bioavailability of P in the sludge and improving the properties of attapulgite in isolation until then [1],[2]. Therefore, their jointuse is justified as a viable alternative due to their effects on the adsorption/desorption of P from sludge by attapulgite for fertilizer in agriculture.

Acknowledgements:
The authors acknowledge à CAPEs.

References:

[1] XU, Chenglong et al. Adsorption and immobilization of phosphorus from eutrophicseawater and sediment using attapulgite-Behavior and mechanism. Chemosphere, v.313, p. 137390, (2023).

[2] LI, Jiawei et al. Enhancing phosphorus bioavailability in sewage sludge through co-hydrothermal treatment with biomass. Journal of Water Process Engineering, v. 51,p.103448, (2023).

Prospective study of advanced materials in optical property

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With the prospective study of advanced materials in optical properties, it is concluded that advanced ceramics is an area with great potential and that has been standing out due to its use in optical applications. Advanced ceramics is a material of great importance in the development of optical devices, being used in several photocatalytic applications. This is an area that presents a high versatility presenting materials with high performance. The objective of this work is to carry out a prospective study of advanced materials, focusing on advanced ceramics, analyzing their optical properties, and thereby identifying trends in research and development, as well as the most relevant companies and research institutions in the area. Based on this, several methods of analysis of literature and articles will be used, including citation search, keyword competition, patent search and collaboration networks. Seeking to identify the main optical properties of advanced ceramics, such as radiation detection. From the analysis of the collected data, it is expected to identify trends in research and development of advanced ceramics focused on the optical part of the material. Based on the results obtained, opportunities for new research and technological developments and the application of advanced ceramics in optical devices in different sectors can be identified. Thus, the prospective study of advanced materials in optical properties is an important and constantly evolving research area, with great potential to contribute to the development of new technologies and optical applications.

Red fluorescent of quantum dots ZnS:Mn powder for visualization of latent fingerprint

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The development of latent fingerprints at crime scenes have been an important step in criminal investigation and forensic science [1]. Fluorescent materials for revealing latent fingerprints (LFP) have increased due to high contrast on several surfaces, sensitivity, and selectivity [2]. In this sense, heavy-metal-free quantum dots (QDs) chemical passivated with polyvinylpyrrolidone (ZnS: Mn/PVP) were synthed via a simple route at room temperature a low-cost to obtain a high luminescence alternative platform for revealing LFP. These revelations have been assayed on four surfaces: glass, ceramic, plastic, and synthetic leather. X-ray powder diffraction (XRD) investigation showed characteristic planes for ZnS structure (cubic crystalline system; space group F-43m) corresponding to the crystallographic data described by Ebnelwaled et al [3]. FTIR spectra for ZnS:Mn revealed a band between 473-535 cm^{-1}related to Mn-O interaction, a signal at 1003 cm^{-1}corresponds to the stretching of the S-O-C bond and stretching of the C-S bond centered at 655 cm^{-1}. Mn^{2+} doped ZnS QDots optical properties exhibited an absorption maximum at 250 nm, witha band gap energy value of 3.13 eV. In addition, ZnS: Mn QDs showed a photoluminescencein the red region with a broad band centered at 590 nm. ZnS: Mn QDs were effective in developing LFP under UV light on different surfaces, being able to identify the pattern of fresh fingerprints developed. Furthermore, the developed images were compared with the control sample, a standard forensic software, and showed an excellent match, suggesting that ZnS:Mn QDs may works a promising low-cost fluorescent powder candidate for forensic applications.

Acknowledgments: LUMIAM, CNPq, CAPES, FAPEAL

References:

[1] J. Lian, F. Meng, W. Wang and Z. Zhang, Front. Chem., 8, 2296-2646 (2020)
[2] C. Xu, R. Zhou, W. He, L. Wu, P. Wu, and X. Hou, Anal. Chem., 86, 3279-3283 (2014)
[3] Z. H. Hamed, K. E. A. Ahmed and H. A. Elsheikh, AUJES, 2, 147-154 (2021)

Removal of nitrate from aqueous solution usingactivated biocarbon

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Concentrations of NO_3^-in groundwater have been increasing, due to the increased agricultural and industrial activity [1]. Due to the high solubility and stability, this ion has a low tendency to be removed by using conventional water treatment technologies such as adsorption. The present work aimed to evaluate the possibility to increase the efficiency of the adsorption process of a commercial biocarbon (C) by the acidic and basic activation. Initially, the C activation test was carried out with 0.5M NaOH and 0.5M H_2SO_4solutions, followed by washing, filtration and drying. Then, affinity tests with activated carbon (AC) were performed using 0.5g of AC in 50 mL of a 50ppm NO_3^-solution. AC affinity studies were performed as a function of the nitrate solution pH (pH 2, 4, 6, 8 and 10). For AC with NaOH no nitrate removal was observed for pH 4, 6, 8 and 10, whereas for pH 2, 83.4% of nitrate was removed. For AC with H_2SO_4, removal of 97.6, 48.9, 21.90, 20.70 and 6.15% were obtained for pH 2, 4, 6, 8 and 10, respectively. The greater efficiency of adsorption in acidic pH of the nitrate solution, mainly in pH 2, is due to the protonation of the active sites of the AC that favor the adsorption of the ionic species NO_3^-. Expanding study, the C activations were carried out with solutions of 0.1, 0.5, 1.0, 2.0, 3.0 and 4.0M H_2SO_4. In the affinity tests with nitrate solution in pH 6,0 were removed 40.4, 41.6, 50.7, 53.0, 55.7, 88.6% of nitrate for AC with solutions 0.1, 0.5, 1.0, 2.0, 3.0 and 4,0M H_2SO_4, respectively. Second Afkhami et al. [2], the treatment of C with H_2SO_4tends to produce positive sites on the C by protonation of surface -OH groups that would cause an increase in electrostatic adsorption of anions ($S\text{-}OH + H^+ \boxed{\leftrightarrow} S\text{-}OH_2^+ / S\text{-}OH_2^+ + X^- \leftrightarrow S\text{-}OH_2X$).

Acknowledgements:
The authors thank CAPES, CNPq, FAPEAL, FINEP.

References:
[1]H. M. A.El-Lateef, et al., Molecules, 27, 2552(2022)
[2]A.Afkhami, T.Madrakian, Z.Karimi, J. hazardous materials, 144, 427(2007)

Reuse of glass waste from photovoltaic panels in the production of geopolymers

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The demand for energy from renewable and sustainable sources is rapidly growing worldwide. In Brazil, hydroelectric energy is the most used source. However, due to the seasonal availability of water resources in some regions, shared use of solar power plants isa viable alternative, both technically and economically. A problem associated with this technology is the high demand for photovoltaic modules and the waste generated during their operation and after their estimated 25-year lifespan, which can reach masses exceeding 50 million tons. Specifically, glass and semiconductor elements can be used in the development of geopolymeric products, partially replacing Portland cement, using aluminosilicate in an alkaline environment. Thus, this investigation aimed to reuse glass and semiconductor material to generate a geopolymer based on metakaolin. The glass and silicon cells were obtained from damaged solar panels during the installation process of the FloatPV SC solar power plant, located in the reservoir of the Santa Clara hydroelectric plant, in the state of Paraná, Brazil. In this case study, damaged photovoltaic panels from silicon polycrystalline photovoltaic modules were used for their mechanical separation. Experimentally, the glass, EVA, and semiconductor material were crushed together in a jaw-type equipment and subsequently milled in a high-energy mill. For their physicochemical characterizations, field emission gun scanning electron microscopy and analysis (FEG-SEM) and EDS, as well as X-ray diffraction analysis, were carried out. The geopolymeric compositions were tested for their mechanical strength according to curing time and temperature. The produced geopolymers showed potential for use in protective coatings for the construction industry.

Reuse of textile waste with nano reinforcement for the development of materials incorporated with nanocomposites

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The use of plastics in Brazil and the world has shown a growing demand due to their qualities, ease of obtaining, and low cost, and, consequently, produces an increase of these plastics in solid urban waste. The destination of these materials, especially in Brazil, is summarized in sending to landfills, incineration and, mechanical and chemical recycling. This study evaluates the possibility of using polyamide textile waste with elastane discarded by the textile industry, as reinforcement for the development of a polymer-clay composite, on a nanometric scale, and develop a recycling route for these materials. In that, the nanocomposite technology adds value to this material. This work used polyamides from textile waste of the industry. These materials were first processed in a compacting machine. Afterward the composites with 1, 2, and 3% in mass of Verde Clara clay/polyamide were prepared in a double screw extruder. The composites were characterized by techniques of X-ray diffraction (XRD), tensile, flexural, impact tests and thermogravimetric analysis (TGA). Results obtained were compared with the properties of the standard samples. As a conclusion of this study, it can be stated that the polyamide textile scraps with elastane can be reused forming nanocomposites with Verde Clara clay. Verde Clara clay in the proportion of 1% already confers gains in the properties of these nanocomposites.

Acknowledgments:
The authors would like to thank CNPq, FAPESP and CAPES for their financial support.

RHEOLOGICAL AND SPECTROSCOPIC STUDIES OF MATERIALS BASED ON GLUTAMATE AND RARE EARTH IONS

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The synthesis of materials containing rare earth ions (RE) is a constantly evolving field in Chemistry. The use of these ions has sparked the interest of scientists due to the wide range of applications related to their electronic, magnetic, and optical properties. Monosodium glutamate (MSG) is a salt derived from glutamic acid, widely used in the food industry. It has been shown that MSG allows for the synthesis of a compound with gel-like behavior when combined with RE ions [1]. The main objective of this work is to expand on the synthesis of this material, and to perform a detailed spectroscopic study of materials based on MSG and RE ions. The methodology used follows that of previous works [1,2] carried out by our research group, which produced transparent, hygroscopic, and highly viscous materials. The 1:1 proportions between the RE (La+3 or Y+3), containing small amounts of Europium (Eu+3), and the ligand (MSG) were chosen for the structure and properties analyses. The infrared and Raman spectra presented similarities in all samples, varying minimally in the spectra of materials containing yttrium. This suggests that coordination between RE ions and MSG occurs similarly. More experiments are under way to better understand the form of interaction between the RE and the MSG. Preliminary rheological studies [2] showed that viscosity and elasticity parameters vary as external stress is applied, characterizing the material as a non-Newtonian fluid. Additional experiments will determine the correct classification of the material.

[1] RIOS, N. P. S. M., AZEVEDO, L. A., NEVES, R. C. S., ALVES JUNIOR, S., FALCAO, E. H. L. Synthesis of luminescent gel-like materials based on glutamate and neodymium(III). Materials Letters, 2018.

[2] SOUZA, D.T.T.O., Study of rheological and spectroscopic properties of materials based on monosodium glutamate and rare earth ions, Graduation monograph, UFRPE, 2021.

We thank CAPES for the support and Professor Wilson B. for his collaboration.

Rheological study of self-aggregated systems composedof different bile salts and lecithin

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Through the development of self-aggregating systems, as well as the determination of the interactions involved in these systems and the understanding of their physicochemical properties, it is possible to elaborate various types of structures depending on the desired application [1]. One way to prepare these systems is by using surfactants, so it is possible to form structures with micelles, reverse micelles, lamellae or liposomes. Aiming at the formation of these structures, lecithin is a natural surfactant composed of two tails in its structure and undergoes self-aggregation in the presence of bile salts [2]. Bile salts are natural amphiphilic surfactants, produced by the liver and their main function is the solubilization of lipids [3]. Thus, bile salt/lecithin systems (sodium taurodeoxycholate (NaTDC) or sodium deoxycholate (NaDC)) in different ratios (0.2; 0.4 and 0.8 (m/m)) wereprepared. The systems were also prepared in the presence of two solutes: propylene glycol or polyethylene oxide, to analyze changes in the packing of aggregates. Dynamic light scattering (DLS), small angle X-ray scattering (SAXS) and rheology analyses were performed to determine the , shape, recombination time and breakage of the aggregates.

Acknowledgments:

CAPES (88887.713913/2022; 0088887.713911/2022-00); FAPESP (22/02092-9; 22/02089-8), Embrapa - SP and Professor Dr. Edvaldo Sabadini (UNICAMP).

References:

[1] Lin, C. Y. and Tung, S. H. Journal of Molecular Liquids. v. 329, 2021.

[2] Tung, S. H. et al. Journal of the American Chemical Society, v. 128, n. 17, p. 5751-5756, 2006.

[3] Cautela J. et al. Colloids and Surfaces A, v. 532, p. 411-419, 2017.

Scalable production of nano-scale Metakaolin and optimal dispersion analysis in cement matrices

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Cement is the most widely used construction material in the world. Nanomaterials are being considered worldwide as a partial filler for cement, especially in the production of high-strength and high-performance cement-based structures [1]. Although the incorporation of some nanoparticles improves the properties of cement based materials, the efficiency of dispersion still remains a significant challenge for nanometric powders. Reaching complete separation of nanoparticles in suspensions or composite matrices is ideal, however, it is difficult due to their high reactivity and aggregation owing to Vanderwall' s forces [2]. This study aims to increase the production of nano-scale metakaolin using the exfoliation method [1], as well as to evaluate its optimal dispersion content in cement matrices. To achieve nanoscale, the mineral platelets must be delaminated and exfoliated. A dispersant solution was prepared by dissolving NH_4Cl powder in distilled water, and metakaolin is added and stirred for homogeneity over 24 hours to ensure exfoliation of the clay plates, then heated and ground into a fine powder. The particles causing exfoliation must be thin and have a large surface area to maximize barrier properties. Organic ammonium chloride meets these criteria and expands the galleries of the clay, causing exfoliation. To confirm the formation of nanoplates and crystallography phase will be performed Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). Zeta Potential (ZP) will be used to verify the suspension stability.

Acknowledgments:

Authors are grateful to CNPq/Brazil (Process 408905/2022-0 and 308353/2022-6), FACEPE (APQ-1251-3.03/22), and CAPES/Brazil (finance code 001) for partially funding this work and scholarship.

References:

[1] M.S. Morsy, H. Shoukry, M.M. Mokhtar, A.M. Ali, S.A. El-Khodary, Constr. Build. Mater. 172 (2018) 243-250.

[2] J.O. Malta, L.V.S. Oliveira, M.M. Ueki, L.S. Barreto, Powder Technol. 383 (2021) 43-55.

Simple fabrication of microfluidics system by do-it-yourself approach for metal oxide synthesis

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Microfluidics (MFs) is a swiftly burgeoning field that deals with the study and manipulation of fluids in small channels, typically in the range of micrometers. Owing to its design, which allows a fine tuning of fluids conditions such as temperature, mixing and reactants concentration, MFs platform can provide an auspicious route to flow synthesis of a myriad of materials. Whilst a settled platform in the synthesis of organic molecules/drugs, microfluidics is yet to fully realize its potential as a tool for the synthesis of nanostructured inorganic materials. In face of this scenario, this study aimed to provide an easy and reliable protocol for producing MFs devices for the synthesis of inorganic nanostructured particles, mainly silicon dioxide (SiO2). For this purpose, MFs devices were produced using a Do-It-Yourself (DIY) approach, which allows researchers to create MFs devices cheaply and easily using techniques like 3D printing, and soft lithography. MFs devices were produced by mold and casting process. First, molds with desired circuit/channel configuration (200-500 μm radius) were produced by stereolithography 3D printing (z resolution of 10 μm). The MFs device upper part was produced by pouring a mixture of poly(dimethylsiloxane) (PDMS) and catalyst over the negative mold, cured and then removed. This system was placed over a flat glass, closing the lower part of the MFs, and attached to an external pumping system. Nanostructured micro particles of SiO2 were produced by the controlled hydrolysis of tetramethylorthosilicate (in ethanol) by water/sodium hydroxide. The resulting particles were characterized by scanning electron microscopy, and the , distribution and microstructure correlated with synthesis temperature, flux and reagent concentration.

Acknowledgment

Thanks to CAPES (88887.671597/2022-00; finance code: 001) for the financial support

Sinterização de filmes cerâmicos usando Laser de CO2

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Thin ceramic films are fundamental in today''s technology, with applications ranging from humidity sensors, optics, and electronics. The manufacturing process of these materials can take various forms. However, most of these processes require high-cost and complex apparatus. A simple, cheap, and easy-to-implement way is the spin-coating technique, which is based on spreading a chemical solution on the substrate as a result of high-speed rotation. After the deposition process, the film must undergo the sintering stage. This stage consists of high-temperature heat treatment and serves both for the elimination of organic matter and for the formation of a solid ceramic layer on the substrate surface. Usually, sintering is performed using a conventional resistive furnace. However, in recent years, there has been growing interest in unconventional sintering processes, such as CO2 laser sintering. Laser sintering has been applied both in the production of solid ceramic bodies and in post-fabrication treatment of ceramic films. These studies show that, in addition to the advantage of being a fast processing method with low contamination levels, laser sintering can induce modifications in the properties of these materials. Moreover, the laser processing of thin ceramic films, in addition to presenting an alternative to the conventional process, also opens up prospects for improving the characteristics of ceramic films and their applications. Therefore, based on these prospects, the present project aims to investigate the feasibility of laser sintering in thin ceramic films and its influence on the physical properties of these films.

Sol-Gel Encapsulation of Enzymes on Silica Based Supports as a Green Biocatalyst for PharmaceuticalsDegradation

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Biocatalytic degradation with the use of enzymes has gained great attention in the last years due to its advantages of high efficiency and environmental friendliness. The major drawbacks, such as low stability and non-reusability have been addressed in different studies and potential strategies were suggested to overcome these limitations. As such, immobilization on different silica based supports provides the possibility of enzyme reuse and prolonged stability in different operational conditions. Previously, a successful immobilization and subsequent removal of different pharmaceuticals, such as acetaminophen and diclofenac in presence of Cd(II) was performed in our group using laccase crosslinking on Fe_3O_4/SiO_2-DTPA hybrid nanocomposites [1]. In another approach, peroxidase enzymes were first immobilized on magnetite nanoparticles, and later encapsulated within a surface silica layer using sol-gel method. The encapsulation of enzymes enhanced their thermal stability and improved the activity over 20 consecutive cycles for 20 days at 55°C[2]. To produce cost-effective and green nanoadsorbents, in our latest work natural silicates were tested as enzyme substrates for core-shell immobilization techniques. With the natural silicate as a core and silica layer as a shell it was possible to encapsulate enzymes for removal and degradation of three different pharmaceuticals: diclofenac, carbamazepine and paracetamol. The biocatalysts demonstrated great oxidation rates for the selected pollutants. The enzyme acted differently in the three chosen supports due to their complex chemical composition which could have effect on overall enzyme activity.

References

[1] I. V. Pylypchuk, V. G. Kessler, G. A. Seisenbaeva, ACS Sustainable Chemistry and Engineering 2018, 6.

[2] I. V. Pylypchuk, G. Daniel, V. G. Kessler, G. A. Seisenbaeva, Nanomaterials 2020, 10, 282.

STUDY OF METALS RECOVERY FROM PHOTOVOLTAIC PANELS THROUGH ACID LEACHING

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The photovoltaic energy is a clean and renewable way to obtain electricity, however solar panels have a lifespan of approximately 30 years, therefore, after this period they need to be correctly recycled [1]. The acid leaching is one of the techniques used in solar panels, where they are crushed into small pieces and submitted to a chemical acid treatment to dissolve the unwanted materials and separate the material of interest. The objective of this study is the recovering of metals by acid leaching. The panel went through the processes of disassembly, comminution in a knife mill, followed by a granulometric separation and finallya magnetic separation. The material was submitted to acid leaching, carried out in three stages, (i) with nitric acid 2 mol/L, (ii) with sulfuric acid 2 mol/L and (iii) with sulfuric acid 2 mol/L and hydrogen peroxide 0.3% in the ratio of 10:1, and the exposure of the material to each leaching step was performed in 30 minutes and 3 hours. Subsequently, the leachates received a solution of potassium ferrocyanide 0.5 mol/L, and a solution of potassium thiocyanate 0.1 mol/L, both of which were analyzed by spectrophotometry in the wavelength region from 250 to 1000 nm. As a result, the granulometric reduction in the mill was successful, allowing adequate reduction of plaque particles. The leachate in contact with potassium ferrocyanide formed the iron ferrocyanide complex, called Prussian Blue. Furthermore, it showed a band at 700 nm, characteristic of this complex. To confirm the presence of iron, the leachate was tested with potassium thiocyanate and the formation of an intense red solution was observed, corresponding to the complex pentaaqua(thiocyanate)iron(III) and presented a band at 480 nm, characteristic of the complex. Therefore, with the results obtained so far, it was possible to identify the $Fe3+$ ions in the panel.

References:

[1] YANG, H.; LIU, J.; YANG, J. Journal of hazardous materials, v. 187, n. 1, p. 393-400, 2011.

Study of the effects of the addition of functionalized graphene oxide on the mechanical properties of the poly(lactic acid) composite

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Polylactic acid (PLA) is a biodegradable polymer that has remarkable properties, e.g., ease of synthesis, high processability and stability, in addition to low cost, thus being a renewable and promising alternative [1]. However, its application is still limited because it has lower mechanical resistance when compared to other engineering polymers [1]. Thus, one of the ways to improve the mechanical performance of PLA is the addition of nanoparticles that promote an increase in the nucleation density, contributing to greater crystallinity of the material [2]. Graphene oxide (GO) has excellent mechanical properties, and has shown a high potential to reinforce polymeric matrices [2]. Because it has a large amount of hydroxyl and carboxyl groups on its surface, it allows the introduction of other functional groups that help in the dispersion and interaction between reinforcement and matrix [2]. The aim of this project is to incorporate functionalized GO into a PLA matrix in order to build a nanostructured polymeric composite with improved resulting properties. GO was functionalized with diamine for subsequent formation of amide bonds through hydrogen bonds from the PLA carbonyl groups. Such interaction is responsible for the nucleation effect and consequent improvement in the crystallinity of the composite. The chemical interaction between the functional groups of the materials was observed using Fourier transform infrared spectroscopy (FTIR). The mechanical performance was evaluated by tensile strength test and dynamic-mechanical analysis (DMA). The determination of the crystallinity of the composite was obtained by X-ray diffraction analysis (XRD).

Acknowledgements:

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001

References:

[1] Delgado-Aguilar, M., et al. 2020. Materials 13(11), 2655.
[2] Zheng, L., et al. 2018. Iran Polym. J 27, 239-252.

Study of the mechanical properties of high initial strength geopolymer concrete

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Through an adequate dosage of the components that constitute the geopolymer, a Geopolymer Cement Concrete (GCC) was developed, which had its characteristics compared with a Portland Cement Concrete (CCP), through the setting of some dosage parameters, such as consumption of binders, water/binder ratio and mortar content [1], [2], [3]. Mechanical tests of compression, diametrical traction, flexion traction, elastic modulus were performed, in addition to the determination of void indices and microscopic analyses. In general, the results of the mechanical characterization of the CCG showed a performance similar to the results of the CCP, with the advantage of developing greater strength at initial ages of 1 and 7 days. At the age of 7 days, the resistance result obtained was about 12% higher than that of the CCP for the same age. In the microstructural analysis of the concretes, it was possible to observe a better matrix-aggregate adhesion in GCC when compared to CCP, probably due to the high densification of the geopolymer matrix.

Acknowledgment:
CAPES and CNPq for financial support, Companhia Siderúrgica de Tubarão for the EGAF donation, and IME's Laboratory of Construction Materials and Soils.

References:
[1] J, Davidovits. Geopolymer Cement. A review, Geopolymer Institute Library. Jan. 2013.
[2] W.P. Zakka, N.H.A.S. Lim, M.C. Khun. A scientometric review of geopolymer concrete. Journal of Cleaner Production, 280 (2021) 124353.
[3] Y. Li, Y. Liu, X. Gong, Z. Nie, S. Cui, Z. Wang, W. Chen Environmental impact analysis of blast furnace slag applied to ordinary Portland cement production J. Clean. Prod., 120 (2016), pp. 221-230.

Study of the optical and structural properties of Titanate nanotubes, TiNTs, exchanged ionically with Er^{3+} and subjected to heat treatment

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Titanium-based nanotubes, TiNTs, were synthed by microwave-assisted alkaline hydrothermal method and subjected to ion exchange reaction with Er^{3+}, ErTiNT ions. The prepared samples were heat treated in an oxidizing and inert atmosphere to investigate the effects of temperature and atmosphere on the properties of these materials. In this work, optical, structural, morphological and crystallographic characterizations such as UV-Vis, PL, Raman, XRD, SEM and TEM were performed. From these characterizations, it was possible to observe that the morphology remained preserved until the temperature of 600 °C, even inthe presence of erbium oxides, which settled on the surface of the nanotubes. With the data obtained by UV-Vis, the band gap value of all samples was calculated and it was found that the variation of the diameter of sodium (Na^{+}) and erbium (Er^{3+}) ions, sodium content and heat treatment influenced the value of the band gap. The strong luminescence was influenced by the amounts of oxygen vacancies, which predominated in the samples heat treated in inert atmosphere. The presence of these defects was determined by the energy of Urbach. Through these studies, it can be suggested the use of materials exchanged with erbium, ErTiNT, in optoelectronic and photonic applications, as photoluminescent devices.

Study of the structure effect on the cloud point of RAFT produced pDMAEMA-b-pS copolymers

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The raising interest in materials for sophisticated applications has driven the development of new methodologies for the production and processing of polymers. Reversible Deactivation Radical Polymerization (RDRP) is among those new concepts [1]. Such techniques allow the synthesis of tailored polymeric materials presenting narrow molecular mass distributions and compositional homogeneity. This is crucial for the study of how average molar mass and composition influence properties. Here, RAFT is used to simultaneously study the influence of aggregation state and the cloud point of PDMAEMA-b-PS copolymers. The thermoresponsive PDMAEMA macroCTA was synthesized using RAFT under GPC monitoring. The reaction presented a steadily molar mass growth up to 30240 g/mol, as expected for a RDRP synthesis. The final Mw/Mn was 1,42. The purified macroCTA was extensively characterized via FTIR and GPC. Cloud point in aqueous media was determined by UV-Vis spectroscopy at different conditions. This macroCTA is to be subsequently used for a copolymerization with styrene to generate block copolymers with different (increasing) PS content, under the exact same conditions. A simultaneous study of aggregation state via DLS and cloud point determination is intended to explore the interrelationship between aggregation and thermal behavior.

Acknowledgements:

CNPq e FAPESP

References:

[1] Moad, G.; Rizzardo, E.; Thang, S. H. Living Radical Polymerization by the RAFT Process - A Third Update. Aust. J. Chem. 2012, 65 (8), 985-1076. https://doi.org/10.1071/CH12295.

[2] Corrigan, N.; Jung, K.; Moad, G.; Hawker, C.; Matyjaszewski, K.; Boyer, C. Reversible-Deactivation Radical Polymerization (Controlled/Living Radical Polymerization): From Discovery to Materials Design and Applications. Progress in Polymer Science 2020, 111, 101311. https://doi.org/10.1016/j.progpolymsci.2020.101311.

Surface free energy evaluation of doped diamond-like carbon films

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Surface free energy (SFE) is critical for understanding adhesion and interfacial interactions in various applications. Anti-scale surfaces, for example, must be engineered to guarantee low chemical affinity with the compounds formed during thermodynamic variations that the contact fluid undergoes. Additionally, surface roughness (SR) can directly influence the wetting behavior of a material, hence, modifying the resultant SFE and playing role into adhesion of the scale. Diamond-like carbon (DLC) films have been increasingly proposed as a coating for applications that demands low adhesion of different products, by tailoring SFE. By adding doping elements, like Silicon and Fluorine, is possible to achieve different ranges of SFE. Moreover, DLC films present other interesting properties as elevated hardness, low friction coefficient and chemical inertia, allowing its application when polymers are not suitable. This study focuses on measuring SFE and SR. A 316L Stainless Steel (SS) substrate was investigated, and three coatings: DLC (reference DLC), DLC-F and DLC-Si. The substrate roughness was also varied, using a polished and a sandblasted finishing. The SFE measurements were conducted via sessile drop contact angle using water, ethylene glycol and diiodomethane as probe liquids. For the SFE measurement, 3 theories were evaluated: OWRK, Wu and Van-Oss. The results show that a decrease in total SFE for the doped-DLC coatings with sandblasted topography, compared to the SS substrate, while the coatings with polished topography showed the opposite behavior. The statistical analysis of the results indicates that, in relation to the SFE methods used, the total SFE converges to average values. When analyzing components of SFE individually a significantly variation of values was observed.

Acknowledgments:
The authors are grateful to ANP for funding and to Labmat-UFSC.

References:

[1] Z. Wang, A. Neville, SPE (2005).
[2] B. Bhushan, Y.C. Jung, Phil. T

Synthesis and characterization of carbon nanomaterials from the mesocarp of babassu coconut, a regional biomass, and its application in the environment

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The activated carbon is characterized by being an excellent adsorbent, besides being non-toxic, and can come from renewable carbonaceous materials [1]. From these characteristics, the biomass from the mesocarp of babassu coconut was used for the synthesis of activated carbon in order to obtain a great porosity and appreciable surface area. Three parameters of synthesis were used: the variation of temperature, the form of pre-treatment and chemical activation with different proportions of potassium hydroxide (KOH), in order to obtain better physical and chemical properties and a better yield. The samples were characterized using techniques such as: Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Elemental Analysis (CHNS), Zeta Potential and the surface area and porosity determined by the BET (Brunauer, Emmett, Teller) method. In order to alleviate the problems caused by textile industries, which produce a large amount of residues, which are often characterized by being carcinogenic and a eutrophication factor, which presents itself as risks to health and to the In the environment [3], activated carbon was applied in the adsorption of the cationic dye methylene blue, so that tests of kinetics and isotherm of adsorption were performed to acquire the relationships of time, concentration and adsorption capacity. About 94% of the tested dye was adsorbed by one of the samples obtained, so that the adsorption would be chemically occurring in monolayers and with a saturation time of 6 hours.

acknowledgements:
UFPI, Capes, Fismat, Limav, PPGCM

References:

[1] MISTAR, Eka Marya; ALFATAH, Tata; SUPARDAN, Muhammad Dani, Journal of Materials Research and technology Vol. 9 6278-6286 (2020).
[2] KHASRI, Azduwi SOLOMON, Olugbenga; AHMAD, Mohd Azmier, Res Chem Intermed, 5737-5757 (2018).
[3]HOU, Jonhua, et al. ACS OMEGA Vol. 5, 13548-1356 (2020).

Synthesis and characterization of iron oxides and their application as inorganic pigment

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Most inorganic pigments are composed of oxides, sulfides, oxyhydroxides, silicates, sulfates, or carbonates, with iron oxides being a special group of natural pigments. Iron oxides have characteristic colors such as red, yellow, brown, green, and black and have good thermal stability, chemical resistance, nanometric , non-toxic and low cost [1]. In this study, synthetic inorganic pigments (SIP) based on iron oxides were synthed by the alkaline precipitation method. The sample was left in aqueous suspension and dispersed in commercial real estate paints, white and colorless, to evaluate their behavior as a pigment. Structural characterization (XRD), composition (EDXRF), spectroscopy (UV-Vis and photoacoustic), and colorimetry were performed. The phases indexed by XRD were goethite, hematite, and magnetite [2,3]. The inorganic pigments produced showed dispersion compatibility in commercial white paint, without changing the surface coating power, and could be an alternative to produce synthetic inorganic pigments and compatible with natural inorganic pigments.

Acknowledgments:
Capes, Fundação Araucária, CNPq, and FINEP. S.J . is thankful for a CNPq PDJ post-doctorate grant (152230/2022-0)

References:
[1] Darbandi, M., Stromberg, F., Fanders, J., Reckers, N., Sanyal, B., Keune, W. Journal of Physics D: Applied Physics,45 ,95001 (2012).
[2] Mariani, F.Q., Borth, K.W., Muller, M., Dalpasquale, M., Anaissi, F.J. Dyes and Pigments 13, 403-9 (2017).
[3] Muller, M., Villalba, J.C., Mariani, F.Q., Dalpasquale, M., Lemos, M.Z., Huila, M.F.G., Anaissi, F.J.. Dyes and Pigments 12, 271-8 (2015).

Synthesis and characterization of MIL-53 (Fe) for removal of organic dyes via the Sono-Fenton Process

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The discharge of persistent organic pollutants (POPs) from wastewater into the environment due to industrial waste is a growing concern [1]. Various methods have been employed for POP elimination, including advanced oxidation processes (AOPs), which generate highly reactive and non-selective hydroxyl radicals (·OH) that can break down pollutants into safer compounds [2]. Among AOPs, the Fenton process is widely used to generate ·OH, often with the aid of other techniques such as ultrasound (known as Sono-Fenton). Metal-Organic Frameworks (MOFs) have emerged as a promising class of heterogeneous Fenton-like catalysts due to their unique crystalline porous structure formed by metallic or clusters connected by organic ligands [3]. In this study, the MOF MIL-53 (Fe) was synthed via the solvothermal method and applied in the Sono-Fenton process to degrade organic dyes (methylene blue and methyl orange). The sample was characterized by XRD, FTIR, SEM/EDS, and TGA. XRD analysis confirmed the formation of desired MOF. The results showed that Fe-MOF is a promising catalyst for selectively removing organic dyes via the Sono-Fenton Process, with a shorter degradation time for methylene blue than for methyl orange with degradation efficiency of 97% and 95,4%, respectively.

Acknowledgments:

The authors gratefully thank CNPq and Capes for the scholarships granted. This work was supported by CNPq (Grants no. 405364/2021-0 and 314590/2021-8).

References:

[1] M. F. Dapaah et al., Chemical Engineering Journal, vol. 431, 134182 (2022)
[2] S. Adityosulindro et al., Ultrason Sonochem, vol. 39, 889-896 (2017)
[3] L. Ferreira Pereira et al., Ultrason Sonochem, vol. 69, 105275 (2020)

Synthesis and characterization of polymeric nanogel containing micronutrient as a potential vehicle for seed germination

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Nanomaterials have become a symbol of great attention in the area of agriculture, due to the continuous technological advance, using nanoparticles and nanocomposites to be used in different aspects and phases of plants, such as planting, nutrition, pest control and genetic improvement (1). In this work, the synthesis of nanogels based on polyetheramine-epoxides was carried out based on literature (2,3). The influence of the amine-epoxide molar ratio on the nanoparticles formation was evaluated. The embedded of Fe ions during the nanoparticles synthesis was studied. All the nanogels obtained were characterized by techniques such as: DLS/ zeta potential, TEM and SEM. Moreover, cucumber seeds was primed with nanogel formulations to studied the shoot/root length. It was observed that the hydrodynamic diameter of the nanogels were 400 nm and 700 nm as a function of metal embedded. The nanogels presenting a negative surface charge in basic medium and slightly positive in neutral or acid medium. Preliminary results, demonstrated that the application of nanogels in Cucumber (Cucumis sativus) seeds, even without the presence of micronutrient, they show a improve of the germiation process. This work open novel perspectives of nanotechnology for agriculture purposes.

Acknowledgements: The authors are grateful for financial support provided by FAPESP (grants #2021/06552-1, #2022/06507-9, #2022/13408-7 and EMU #2021/14619-9), CAPES (Finance Code 001), and CNPq (grant #307696/2021-9).

References:

[1] B. Ndaba, A. Roopnarain, B. Vatsha, S. Marx, M. Maaza, ACS Agricultural Science Technology 2., 1218-1229 (2022)

[2] S. Tang, L. Hang, Z. Shi, W. He, J. Mater. Chem. B, 3., 2842-2852 (2015)

[3] S. Tang, L. Huang, R. J. Daniels-Mulholland, E. Dlugosz, E. A. Morin, S. Lenaghan, W. He, Acta Biomater, vol. 43, 292-30 (2016)

Synthesis and Characterization of $Sr_{1,75}Ca_{0.25}Fe_{1,5-x}Ni_xMo_{0,5}O_{6-\delta}$ with Doping by Mechanosynthesis and Production of Nanoparticles by Exsolution Aiming Application in Symmetrical Solid Oxide Fuel Cells

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Nowadays, the need to produce clean and renewable energy is increasing. Solid Oxide Fuel Cells emerge as a strong candidate, converting chemical energy into electrical energy, without the need for combustion, releasing almost no toxic substances into the environment. Symmetrical solid oxide fuel cells (SSOFCs) use the same material for both electrodes, having advantages over common cells such as reduction of difficulties in chemical and thermal compatibility between the electrodes and the electrolyte. This work presents the synthesis of $Sr_{1,75}Ca_{0.25}Fe_{1,5-x}Ni_xMo_{0,5}O_{6-\delta}$ (SCFNMO) aiming to transform CH_4 into syngas directly in the SOFC anode chamber. $Sr_2Fe_{1,5}Mo_{0,5}O_{6-\delta}$ (SFM) is promising for application in SSOFCs, as it proved to be stable in oxidation and reduction reactions [1]. To improve the characteristics of SFM, A and B sites can be doped with calcium and nickel, respectively, promoting better electronic conductivity and increasing the catalytic activity in the dry reform of CH_4. Three different compositions will be studied: $Sr_{1,75}Ca_{0.25}Fe_{1,25}Ni_{0,25}Mo_{0,5}O_{6-\delta}$, $Sr_{1,75}Ca_{0.25}FeNi_{0,5}Mo_{0,5}O_{6-\delta}$, $Sr_{1,75}Ca_{0.25}Fe_{0,75}Ni_{0,75}Mo_{0,5}O_{6-\delta}$. SCFNMO powders were synthed by chemical route (Pechini method), followed by heat treatment. After that, $Sr_{1,75}Ca_{0.25}Fe_{1,5}Mo_{0,5}O_{6-\delta}$ powders are going to be synthed by the chemical route and will be doped with NiO by mechanosynthesis, using planetary ball mills. In future developments, reduction treatments will be performed to obtain Ni nanoparticles through exsolution in the matrix and catalytic tests will be performed. Detailed characterization of the powder will be carried out by means of X-ray diffraction, simultaneous thermal analysis, dilatometry and scanning electron microscopy.

Acknowledgements:
National Oil Agency (ANP), CINE-SHELL/ FAPESP grant n. 2017/11937-4, 2018/04596-9 and 2018/04595-2

References:
[1] Q. Liu, D. E. Bugaris, G. Xiao, M. Chmara, S. Ma, H. Z. Loye, M. D. Amiridis, F. Chen, Journal of Power Sources 2011, 196, 9148-9153.

Synthesis and characterization of the β-LiAlSi2O6doped with Mn and Cu to ceramic pigment.

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Ceramic pigments are considered as a group of materials that impart color (DIMITROV et al., 2017). Several colors can be found in pigments and they depend on the addition of the dopant and method of production (ZAICHUK; AMELINA, 2017). Considering the new ceramic materials, there is spodumene (LiAlSi2O6). The most common means of productionis the solid-state, but the high temperatures used, high production cost and problems in homogenization (SEDEL' NIKOVA et al., 2011) are obstacles encountered. The present work proposes the synthesis and characterization of the β-LiAlSi2O6 doped with Mn and Cu to study the relationship between the structure and color of pigments. The samples were prepared by proteic sol-gel method, using the proportion 1:0,5 of reagents:gelatin. A solution was prepared with gelatin and water at a temperature of 70 ºC and constant agitation. Then the following reagents were added: aluminum nitrate nonahydrate, lithium nitrate, silica oxide and the dopants $Mn(NO_3)_2$ and Cu(NO3)2 in different concentrations(2%, 2,5% and 3% in mass). The final solution was then heated to a temperature of 200 °C,for one hour to form a gel. The gel was dried at 100 ºC by 48 h. After drying, the xerogelwas crushed and then subjected to pre-calcination, of 700 °C by 2h. Then the samples werecalcined for two hours at 110 ºC and cooled freely. X-ray diffraction (XRD), scanning electron microscopy (SEM) and X-ray fuorescence (XRF) were performed. XRD results confirmed that the cryst structure of lattice correspondend to β-spodumene, compatible withreference ICSD 14235. SEM analysis identified polycrystalline particles of micrometric order, with irregular morphology for all samples. XRF analysis identified the elemental composition of the samples. Preliminary results show the viability of the synthesis of β-LiAlSi2O6 through the protein sol-gel method and a great potencial for obtaining ceramic pigment in different shades of blue and gray.

Synthesis and modification of deep eutectic solvents (hDES) for removal of inorganic contaminants in sewage sample

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A stream of studies called Green Analytical Chemistry was initiated in recent decades with the aim of reducing or eliminating the use of toxic products for humans and the environment [1]. In this context, the deep eutectic hydrophobic solvents (hDES) has been highlighted by particularities such as biodegradability and high extraction efficiency [1]. The aim of this study was to prepare a hDES and modify it with Fe_2O_3 particles to apply it in the removal of inorganic contaminants in sewage samples. The first step involved the preparation of the hDES from a mixture of acetic acid and DL-menthol (1:1 molar ratio) under stirring at 200 rpm and heating for 15 min at 60 °C. The physical-chemistry properties of the formed solvent were obtained by density and viscosity measurement and spectroscopic characterization by ATR-FTIR mode. The interpretation of the main vibrational modes present in the FTIR spectra of the precursors and hDES showed a shift of the symmetric stretching of the carbonyl group (υ_sC=O) from 1704 to 1713 cm^{-1}, indicating the formation of hDES. The second step involved the hDES modification by the Fe_2O_3particles, prepared by coprecipitation from ions Fe^{2+} and Fe^{+3}solution in pH 9.0. Structural studies from X-Ray Diffraction confirmed the formation of maghemite (γ - Fe_2O_3) crystalline phase (PDF25-1402/ cubic, space group ($P4_32_12$), a = b = c = 8.34 Å). The morphological study of γ - Fe_2O_3 particles from Transmission Electron Microscopy showedan average distribution lower than 500 nm. The next step will focus on the use of hDES modifiers with Fe_2O_3 particles in the removal of Cd and As present in sewage samples and ICP-MS analysis.

References

[1] C. Florindo, et al, Journal of Molecular Liquids 297,111841 (2020).

Acknowledgments

São Paulo Research Foundation (FAPESP grants #2014/50945-4, #2021/14581-1 and #2021/14759-5), CAPES, CNPq, IBILCE-UNESP-São José do Rio Preto and INCT-DATREM.

Synthesis, Characterization, and Microbial Activity of Quercetin-Conjugated Simonkolleite Nanocrystals

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Extensive research has demonstrated that quercetin has potential health benefits, including mitigating inflammation and oxidative stress, which are critical factors in the development of numerous chronic diseases such as cancer, heart disease, and neurodegenerative disorders. Conjugating quercetin with nanocrystals is an innovative technique with tremendous potential that improves its solubility, bioavailability, bioactivity, and therapeutic potential. Additionally, it can improve the stability of quercetin and protect it from degradation while enhancing its imaging capabilities by providing contrast in biological samples. The group has expertise in the synthesis and characterization of biocompatible nanoparticles [1] and recently of simonkolleite nanocrystals,[2] which has few works in the medical and biological area. Therefore, in this study, simonkolleite nanocrystals were conjugated with quercetin and to investigate the conjugation infrared spectroscopy, and scanning electron microscopy were used. The results confirmed the successful conjugation. Moreover, the microbiological assays revealed that the conjugated nanocrystals had significant antimicrobial activity against various microorganisms, including Gram-positive and Gram-negative bacteria as well as fungal strains. As a result, simonkolleite nanocrystals hold promise as a material for various biomedical applications due to their enhanced stability, biocompatibility and the ease of conjugation with quercetin to potential for medical applications.

ACKNOWLEDGEMENTS: CNPQ, FAPEAL and CAPES.

[1] A. Silva, Anielle et al. Doped Semiconductor Nanocrystals: Development and Applications. Nanocrystals.: IntechOpen, 2021, 1;

[2] A. Silva, Anielle et al. Synergistic effect of simonkolleite with zinc oxide: Physico-chemical properties and cytotoxicity in breast cancer cells, Materials Chemistry and Physics, 266, 2021, 124548, 0254-0584.

Synthesis of activated charcoal from Uxi seed and Scanning Electron Microscopy and Energy Dispersive Spectroscopy Characterization

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The species Endopleura uchi, Humiriaceae family, native to the Amazon, is commonly known as uxi-amarelo or uxi-liso [1, 2]. This fruit has many applications in diverse areas, aiming to contribute to sustainable development. This study presents to synthesis of activated charcoal from the uxi fruit endocarp, using physical activation (calcinations temperature) and chemical activation. The uxi fruit material was collected at the Ver-o-peso fair, the pulp was removed and were used only the seeds. The seeds were dried at room temperature and after in an oven at 60 °C, they were fractionated and then taken to the millfor maceration. The samples were sieved with a granulometry of 250 mm/ · m and was chemically activated with phosphoric acid and ferric chloride. The solution was agitated for 24 h on a magnetic stirrer and after filtered for 24h. The solution was agitated for 24 h on a magnetic stirrer and filtered for 24h. After drying, the obtained material was calcined at 700 °C for 2 h. The same calcination process was carried out for in natura sample. In orderto observe porosity, morphologically characterization was carried out on these two uxi samples by SEM (Scanning Electron Microscope) and also EDS (Energy Dispersive Spectroscopy) characterization. Scanning Electron Microscopy (SEM) images indicated thepresence of pores with an average size of 1.6 μm. From the obtained material, adsorptiontests can be carried out, in order to purify water.

Synthesis of a renewable polymeric material derivedfrom glycerol to be applied as an adsorbent to dyes

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Polymers are extensively used in the world and most of them are from non-renewable sources. Then, sustainable sources must be founded to produce these materials. Due to the interesting resulting properties, the amines are deeply used as monomers to produce polyurea, polyamides, and others [1]. Therefore, they can be used as a crosslinking agent [2] to produce biopolymers when reacted with a pre-polymer based on glycerol and maleic anhydride. In this sense, this work produced a polymeric material based on the thermal polymerization of these reactants (235 °C for 10 minutes without using catalyst). In the mid-infrared absorption analysis (MIR) was observed changes in the spectra, corroborating the occurrence of polymerization. The final polymer presented interesting adsorbent characteristics when in contact with rhodamine B, exhibiting a potential for use as a wastewater decontaminant.

Acknowledgments: FAPESP (grants 2022/03489-0 and 2021/02152-9) and CNPq (grants 303247/2021-5 and 150233/2022-1).

References:

[1] V. Froidevaux, C. Negrell, S. Caillol, J. P. Pascault, B. Boutevin. Chemical Reviews, v. 116, p. 14181-14224 (2016).

[2] S. S. Gupta, V. Mishra, M. D. Mukherjee, P. Saini, K. R. Ranjan. International Journal of Biological Macromolecules, v. 188, p. 542-567 (2021).

Synthesis of biochar dopped with nitrogen by wet route: effect of nitrogen concentration in the dye adsorption capacity.

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During the production of sugar and ethanol several solid products are obtained, among them, a fraction of biomass[1]. In this work, various conditions to favour the obtaining of a high surface biochar were studied, using waste biomass of the sugar and alcohol industry as raw material. After granulometric analysis, two fractions of biochar with different granulometries were purified and dopped with nitrogen by wet route for later study of the adsorption capacity and selectivity, using methylene blue and methyl orange. Granulometric analysis showed a higher proportion of carbonaceous material with diameter above 600 μm. After purification process, Thermogravimetric Analysis showed that the solids presented 2 to 3% of inorganic material. The inorganic fraction is formed mainly by silicon, probably in the form of silicon oxide. The precursor biochar with diameter above 600 μm presented a B.E.T. surface area of 482 m^2/g and a mono-modal distribution of pore with an average of 47 Å, calculated by the BJH method.

Acknowledgments: The authors acknowledge the support of: Laboratório de Síntese de Catalisadores, LSCat - UFAL, Laboratório Tecnano - UFAL

References:

[1] SANTOS, F. et al. Sugarcane: Agricultural production, bioenergy, and ethanol. Elsevier Inc, 2015.

Synthesis of *m*-phenyldiaminadansil to obtain carbondots for application as OFF-ON pH sensor.

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Carbon dots (C-dots) are luminescent biocompatible carbon nanoparticles, which may be obtained from several precursors, including organic molecules[1]. They have been applied as optoelectronic devices, fluorescent anti-counterfeiting inks and fluorescent probes. C-dots are promising as pH sensors due to their easy preparation, highly stable fluorescence, low toxicity and low cost[2]. Therefore, *m*-phenyldiaminedansyl (*m*-PDDS) was synthed and applied to obtain C-dots (C*m*PDDS) that were evaluated against pH variation. The m-PDDS was obtained from a nucleophilic acyl substitution reaction with dansyl chloride in the presence of triethylamine and chloroform as solvent, with 60% yield and was characterized by ^{1}H NMR, ^{13}C NMR and FTIR. Subsequently, 10 mg (0.032 mmol) of *m*-PDDS were subjected to hydrothermal synthesis with 10 mL of distilled water in a reactor at 200°C for 4 hours to obtain the C*m*PDDS, which were centrifuged at 13,000 rpm for 10 minutes and filtered through a membrane of 20 µm. The C*m*PDDS obtained exhibited a smaller than 10 nm, with an interplanar spacing of 0.23 nm, characteristic bands of S and N groups, UV-Vis bands in the region of 257 nm and the range of 281 to 447 nm of C=N and C-N, and demonstrated an excitation wavelength-independent behavior with maximum emissions at 478 nm when excited at excitation lengths of 440 nm. The fluorescence behavior of CmPDDS was evaluated against different pH values. These exhibited an OFF-ON behavior with increasing pH (2-12), demonstrating the potential to be applied as an OFF-ON pH fluorescent sensor.

Acknowledgment:
To the funding agencies CNPq, CAPES e a FAPEAL.

References:

[1] WU, W., LIU, H., et al. Chemical Communications, 57, 40, 4894-4897, 2021.
[2] DAVI, LBO, LIMA, DJP, BARBOSA, CDAES. Optical Materials, 121, 111502, 2021.

Terahertz spectroscopy characterization doped n-type AlGaN thin films

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In this study, we investigate a series of $Al_{0.67}Ga_{0.33}N$ ternary alloys with varying silicon n-type doping levels using terahertz time-domain spectroscopy (THz-TDS). We investigated the impact of silicon doping on THz response, observing a continuous absorption increase until saturation at high concentrations above $10^{19} cm^{-3}$, then a decrease resembling absorption levels of the undoped samples, indicative of a compensation regime. A similar trend was identified through specific conductivity measurements. These findings suggest that THz- TDS can determine the optimal silicon doping level for AlGaN thin films to enhance their electronic transport properties.

THERMAL PROPERTIES OF EPOXY-ACID RESINS

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Technological advances have provided many researches to produce high performance epoxy resins to be widely applied industrially in coatings, adhesives, insulators, etc. [1]. Epoxy-based thermosetting resins can be produced by the reaction between diepoxy compounds and organic molecules, resulting in organic networks with a high mechanical strength and good thermal stability. [2]. This study aimed to evaluate the thermal properties of epoxy resins cured with different structures of carboxylic acids, citric acid (tricarboxylic acid), and sebacic acid (dicarboxylic acid). Stoichiometry of 1:1 (COOH/epoxy) molar equivalent, 130 °C for 72 hours, were used as parameters in the curing of the epoxy resin. The FT-IR spectra of the epoxy-acid resins were evaluated to verify whether there was a complete curing reaction and identify the main functional groups present in the studied samples. The thermal properties were evaluated by Differential Scanning Calorimetry (DSC) tests to verify whether there was a displacement of the glass transition temperature, and the thermal stability of the cured resins was evaluated by Thermogravimetric Analysis (TGA). Asa result, it was observed that the use of acids with different chain s influenced the variationin the final structure of the polymer and its thermal stability; the resin cured with sebacic acid, owing to its linear structure, presented lower thermal stability and glass transition than those cured with citric acid.

Acknowledgements:
Work supported by CNPq.

References:

[1] XIE, Meira WANG, Zhonggang; ZHAO, Yunfeng. Synthesis and properties of a novel, liquid, trifunctional, cycloaliphatic epoxide. Journal of Polymer Science Part A: Polymer Chemistry, v. 39, n. 16, p. 2799-2804 (2001).

[2] CUNHA, Rodrigo H.; NELE, Marcio; DIAS, Marcos L. Reaction and thermal behavior of vitrimer-like polyhydroxy esters based on polyethylene glycol diglycidyl ether. Journal of Applied Polymer Science, v. 137, n. 43, p. 49329 (2020).

Thermometric properties of Nd-doped BaTiO3 particles operating in the first biological window

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The investigation of contactless luminescent thermometers imposes the search for new materials with increasingly better thermometric properties, as well as its obtaining method [1]. The ease in modulating the spectroscopic properties of materials doped with lanthanide ions makes them particularly attractive for this type of application. In this work, barium titanate particles (BaTiO3) doped with Nd3+ were synthed by a new eco-friendly sol-gel route. In this route, the chelating capacity of the natural organic matter contained in river water was used to polymerize the metal solution, which is then calcined, resulting in crystalline nanoparticles. Parameters such as concentration, pH and calcination temperature were investigated during production, with the purpose of developing a simple, efficient, and low energy cost procedure. Samples with Nd3+ molar concentrations of 0.5, 1 and 3 mol% were produced. Crystallite of 22 nm was determined from XRD data using Scherrer´s equation. SEM images revealed particles in the form of overlapping plates. The technique used for the thermometric characterization of the samples was based on the peak valley intensity ratio under excitation of 800 nm. The high relative sensitivity (Sr) values of Nd-BaTiO3 particles produced indicate the potentialities of these materials for temperature detection on nanoscale. In addition, from the results of repeatability tests, it was possible to observe that this material has excellent stability in the temperature range from 303 to 373 K.

Acknowledgment: This work has been supported by the Brazilian funding agencies CNPq, CAPES, FINEP and FAPITEC. This research used facilities of the CMNano/UFS under proposal #080/2023.

References:

[1] N. Rakov, Y. Xing, G.S. Maciel, Optical thermometry operation within all three biological windows using Nd3+:Er3+:Y2O3nanocomposite phosphors, ACS Appl. Nano Mater. 3 (2020) 10479-10486. https://doi.org/10.1021/acsanm.0c02397.

The use of biomass residues for the preparation of luminescence interference adsorbents

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Activated carbon (AC) has been used as an adsorbent to remove contaminants, mainly due to its high surface area, pore volume, and surface functional groups. The use of biomass as precursors have been increasing due to its abundantly available, low cost, and environmental impact. In this study, were prepared ACs from sugarcane bagasse and coffee husk, impregnated with phosphoric acid in 1:1 and 1:3 (w/w) ratios, and activated by microwave oven times of 10 and 20 minutes and heating in 500 and 1000 W. The produced AC was used to remove iron and copper ions in aqueous solutions, which are luminescence interferents. The ACs were analyzed by adsorption/desorption isotherms to calculate the apparent surface area by BET equation. The AC from sugarcane bagasse prepared within 20 minutes and 1000 W has a surface area and pore volume of 1055 $m^2. g^{-1}$and 0.511 $cm^3. g^{-1}$, respectively. The AC from husk coffee obtained had the lowest time and higher heating (10 minutes and 1000 W), showing a surface area of 708 $m^2. g^{-1}$and volume total of pores of 0.619 $cm^3. g^{-1}$. Both AC prepared with higher amounts of activating agents showed the presence of mesopores. Iron and copper complexes in different concentrations were prepared for the adsorption tests, and europium and terbium lanthanide complexes were used as luminescent sensors. Firstly, adsorption experiments of iron were made with commercial AC. The final ion concentration was determined using a calibration curve (intensity ratio for $[Eu(DPA)_3]$ with added $FeCl_3$), and was observed a decrease in the luminescence intensity as a function of iron concentration. The equilibrium conditions were obtained in 24 hours, with 10 mg of AC and 10 mL of iron solution. Under these conditions, 30% and 10% were removed for the solution containing 50 and 125 $mol.L^{-1}$of iron, respectively.

Acknowledgments:
The Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and FAPESP (2019/13471-8).

The use of rate equations to model the emissions of sodium niobate nanoparticles co-doped with ytterbium and thulium ions

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Nanoparticles (NPs) doped with rare earth ions have immense potential for various applications, including biology, medicine, and the development of solar cells. Among the diverse areas of rare earth-doped NP research, nanothermometry has gained considerable attention [1,2]. This is partly due to its relevance to biological applications, where the precise determination of local tissue temperature can be a valuable tool in the early detection of inflammation and tumors, including cancer [3]. To contribute towards the development of more efficient thermometric systems, we conducted a theoretical and computational study to investigate the key factors that affect the emissions and relative thermal sensitivity of a system (Sodium Niobate NPs) co-doped with Ytterbium and Thulium under excitation at 1064 nm. To achieve this goal, rate equations were employed to model the emissions of this system. As a result, we found that the variations in emissions with temperature were consistent with the experimental results, which validated our model. The fluorescence intensity ratio (FIR) between the emissions at 480 and 800 nm was used to calculate the relative thermal sensitivity, whose maximum value was equal to 0.7%/K at 450 K.

Acknowledgments
We acknowledge the financial support from Brazilian Agencies: FINEP (Financiadora de Estudos e Projetos) through the grants INFRAPESQ-11 and INFRAPESQ-12; CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnologico) through the grants: Projeto Universal Nr. 431736/2018-9 (C. Jacinto); CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico) by means of the PIBIC/2022-2023 scholarship of J. P. G. Almeida.

References
[1] B. del Rosal, E. Ximendes, U. Rocha, and D. Jaque, Adv Opt Mater 5(1), (2017).
[2] D. Jaque and F. Vetrone, Nanoscale 4(15), 4301-4326 (2012).
[3] I. Brigger, C. Dubernet, and P. Couvreur, Adv Drug Deliv Rev (2012).

TOPOGRAPHIC CRITERIA TO OBTAIN SUPERHYDROFOBICITY

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Superhydrophobic surfaces have the property of repelling water, these materials have interesting features, anti-corrosion, self-cleaning, anti-contamination, among others. These characteristics are important to the industry in general. Barthlott's studies [1] on the lotus effect show that a superhydrophobic surface is composed of low surface energy allied with a nano or microstructured roughness. However, many surfaces structuring processes are expensive and complex. With the motivation of decrease the cost of production and provide new routes for the production of superhydrophobic surfaces this theoretical-experimental work proposes a topographic criteria to obtain superhydrophobicity. With this objective, thin alumina films with graduated roughness were tested. The synthesis of the material was carried out through the sol-gel process, the films were deposited using dipcoting and texturized with chemical etching with boiling water. The low surface energy was achieved through the deposition of a coat of Dynasylan F881. The wetability of the films was analyzed through hysteresis and contact angle measurement and atomic force microscopy was used to characterize the topography. A theoretical equation for the contact angle for the case of heterogeneous surface wetting in terms of the slope of the structures was proposed and compared with the experimental results. The results show that we obtained nanostructured films and increase in etching time tends to increase the roughness and slope of the surfaces. The contact angle was increased with increasing etching time. It was possible to reach the static and dynamic criteria for superhydrophobicity ($\theta \geq 150°$ and $h \leq 10°$). The experimental data showed the same behavior as the theoretical equation, so it was possible to define aslope value to obtain the static criterion for superhydrophobicity, σ_SH=1,61. [1] Wi. Barthlott; C. Neinhuis, Planta, v. 202, n. 1 (1997), 1-8.. The authors acknowledge to CNPq,CAPES and Fapergs.

Transparent glass-ceramics containing $TbPO_4$ crystals:a promising material for photonic and magneto-optical applications

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Transparent glass-ceramics have been a subject of continuous research due to the possibility of in-situ growth of rare-earth single crystals using the glass as a host matrix [1]. Glass-ceramics based on a composition of GeO_2-$SbPO_4$-Al_2O_3-Tb_4O_7 (GSA:Tb) have been studied. This study reports the thermal, structural, morphological and spectroscopic analyses of GSA:xTb glass-ceramics, where x ranges from 0.2 to 0.7 mol% of Tb_4O_7. Differential Scanning Calorimetry (DSC), X-ray Powder Diffraction (XRD), SEM-EDS mapping and Photoluminescence (PL) spectroscopy were used for the analyses. DSC curves for all samples indicated the presence of the glass transition phenomenon and the XRD patterns of the GSA:xTb samples showed amorphous halos and crystallization peaks, confirming the vitreous nature of all glass-ceramics analyzed. XRD patterns of GSA:xTb glass-ceramics with x = 0.5, 0.6, and 0.7 mol% of Tb_4O_7 displayed diffraction peaks attributed to the crystalline phase of $TbPO_4$. The $TbPO_4$ crystals embedded in the volume of the glass-ceramics exhibited an average distribution ranging from 10-100 µm. SEM-EDS mapping of the crystal surface indicated that it is composed of P, O and Tb elements. The $TbPO_4$ crystals displayed a novel morphology and unique design resembling an "H" shape. The PL spectra of GSA:xTb showed emission bands at 491, 548, 588 and 623 nm assigned to the electronic transitions from the $^7F_6$ ground state to the excited levels ($^5D_4 \rightarrow {}^7F_6$), ($^5D_4 \rightarrow {}^7F_5$), ($^5D_4 \rightarrow {}^7F_4$) and ($^5D_4 \rightarrow {}^7F_3$), respectively. The GSA:xTb glass-ceramics have great potential for the development of new Faraday rotators for detecting magnetic fields in industrial electrical systems in the future.

Acknowledgments

The authors are grateful to the São Paulo State Research Foundation (grants 2016/16900-9, 2018/19272-4 and 2023/04832-2) for financial support.

Reference

[1] JUÁREZ-BATALLA, J.; MEZA- ROCHA, A. MUNOZ, G.; CAMARILLO, I.; CALDINO, U.; Opt. Materials, 58, (2016), 406-411.

ULTRA-FAST PRODUCTION OF YF 3 : INFLUENCE OF THE SYNTHESIS PARAMETERS ON THE SAMPLE PROPERTIES

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This work investigates the properties of YF3 produced via microwave-assisted hydrothermal route [1]. The correlation between synthesis parameters and structural, microstructural, and optical properties was explored, to maximize the performance, while reducing the costs and the environmental impact of production. For the synthesis, the starting reagents NH4F and Y(NO3)3 were used in stoichiometric proportion. Reactions were conducted in a sealed Teflon autoclave, under microwave heating, at 140 ºC and autogenous pressure. Different soaking times were tested (1, 5, 15, 30 and 60 min). After the synthesis, the samples were washed and dried. X-ray powder diffraction (XRD) showed that YF3 crystalline phase was already obtained after 1 min of processing. The XRD peak widths vary according to the synthesis time, indicating a dependency of crystallite on the soaking time. Scanning Electron Microscopy (SEM) images presented rod-like morphology for all the samples produced. Preliminary XPS measurements revealed a dependency between oxygen content and processing time of the samples. The dependency of relative sensitivity of Nd-doped YF3on the processing parameters is currently being investigated, for application in non-contact optical nanothermometry.

Acknowledgments: The authors acknowledge CAPES, CNPq and FAPITEC for the scholarship, CMNano-UFS and LNNano for the facilities.

[1] CRUZ, B. M. et al. One-step synthesis of YF3: Nd rod-like particles for contactless luminescent thermometers. Optical Materials, v. 131, p. 112661, 2022.

Umbilical Cable Outer Sheath Service Life Estimation Through Thermal Ageing Tests

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Submarine Umbilical Cables are complex structures which work on a critical and adverse scenario on the seabed. The Umbilicals are composed of structural layers and functional components with many different functions when connected to the subsea production system. This paper presents an accelerated ageing test method to evaluate the behavior of high density polyethylene (HDPE) material, which is applied in the outer sheath of the umbilical cables. This methodology will present a mathematical model to estimate the useful life of HDPE material in operation, using the Arrhenius Equation. The methodology consists of accelerated ageing of the HDPE samples at predefined temperatures for a determined period that will be enough to occur the decrease of mechanical properties. The specimens were stamped of the samples removed from the outer sheath of the umbilical cable after extrusion process. A quantity of 220 specimens of type V were prepared according to ASTM D-638 and they were placed in ovens with specific temperatures (100, 110 e 125°C). The test was periodically monitored through variation of the mechanical properties of the material. The results of the mechanical properties of the aged samples were compared with the results of the unaged samples, and with the correlation of the temperature versus time was possible develop a mathematical model capable of predicting the lifetime of the material. After all test performed, we confirm that the HDPE is a material with excellent mechanical strength, and according to the Arrhenius Plot, this material is considered suitable for production the outer sheath because it has durability greater than 30 years at an operational temperature of 85°C.

References:

[1] HAN, Seong Ok; LEE, Dong Wo HAN, Oc Hee. Thermal degradation of crosslinked high-density polyethylene, 1999.

[2] OLIVIER, Nelson. Análise de falha da camada polimérica externa de cabos umbilicais, 2007.

[3] ISO 13628-5 – Second Edition – December 15, 2009

Use of cementitious composite in bridge pavement reinforcement: The addition of superabsorbent polymers (SAPs) and fibers in ultra-high-performanceconcrete (UHPC): a review study

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Ultra-high-performance concrete (UHPC) is a promising material for repairing rigid bridge pavements due to its dense and refined microstructure resulting from a high cement and fine materials content and low water/binder ratio. However, UHPC is prone to shrinkage cracking, especially due to autogenous shrinkage. To mitigate this, fibers are often added to improve the material''s tenacity and minimize crack propagation. Additionally, the addition of superabsorbent polymers (SAP) has been proven effective in reducing shrinkage strain in cementitious materials through internal curing [1].Both fibers and SAP complement each other in reducing the risk of shrinkage cracking in UHPC, and recent studies have investigated their combined use. This study aims to analyze recent developments in the synergistic effect of adding metallic and synthetic fibers with SAP in UHPC applied in bridge deck pavements. The research focuses on identifying knowledge gaps in the use of non-conventional fibers for pavement reinforcement. A bibliographic search conducted on ScienceDirect found that UHPC with steel fibers and SAP has been used in studies on rigid pavements. However, the use of UHPC for reinforcement and polymeric fibers is not common. High-modulus polyethylene (HMPE) is a potential alternative because of its resistance to severe environments and low weight. The combination of SAP and HMPE fibers could achieve a synergistic effect to improve the efficiency of reinforcement by reducing shrinkage and improving cement hydration. This study highlights a knowledge gap in the combined use of SAP and fibers, particularly HMPE, which could provide an innovative solution for conserving roads/bridges.

Acknowledgments:
The authors would like to thank the UFAL and IFAL for the financial assistance in the study.

References

[1] MECHTCHERINE, V. et al. Application of super absorbent polymers (SAP) in concrete construction-update of RILEM state-of-the-art report. Materials and Structures, v. 54, 2021.

Use of humic substances to obtain δ and γ alumina in Ni/Al_2O_3 catalytic materials

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Humic Substances (SH) consist of 70% to 80% of organic matter and one of their main characteristics is the high complexation with metallic ions [1,2], promoting excellent metallic dispersion in catalytic supports. Thus, this work proposes the use of SH as complexing agents to obtain nickel anchored on alumina (Al_2O_3). The materials were synthed using a simple technique, wet impregnation in the presence and absence of SH. 5, 10 and 15% of Ni relative to alumina (m/m) were used, calcined at 700 °C, followed by characterization by thermogravimetry (TG), X-ray diffraction (XRD), Fourier transform spectroscopy (FTIR) and liquid N_2 adsorption/desorption at 77K and thermoprogrammed reduction (TPR). The materials synthed in the SH presence showed alumina in δ the γ and phase, lower nickel oxides reduction temperature and higher surface area. In preliminary tests in the methane decomposition to obtain ordered carbon and H_2, the catalysts synthed in the presence of SH showed superior catalytic activity.

Acknowledgements: CNPQ, CAPES, FAPEAL for funding the research, UFAL, GCaR, TECNANO/UFAL for XRD analyses, GON for FTIR analyses, and LSCat for TGA analyses.

References:

[1] J.C. Raposo; U. Villanueva; M. Olivare J.M. Madariaga, Microchemical Journal. 128, 26-33, (2016).

[2] W.G. Botero, Humic Substances: interactions with nutrients and contaminants, São Paulo, Brazil, (2010).

Using Advanced Ceramics in Photoluminescence

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Advanced ceramics, materials with exceptional properties, such as strength, high melting points and electrical insulation, stand out for their degree of innovation and their applications. The main objective of this study is linked to highlighting the positive properties of these materials in photoluminescence, a phenomenon responsible for the absorption of light in the material in a certain wavelength range, emitting light in response. The methodology employed consisted of prospecting search with specific keywords, by journals, and patents, showing that China has played an important role in the research and development of advanced materials in recent decades. Between 2012 and 2022, there was a considerable increase in investment in research in this area. Highlighting nickel tungstate, used as an example of a material with interesting photoluminescent properties, being useful in significant technological advances for understanding the fundamental principles of synthesis and processing of advanced materials, which may have important implications for other areas of research in materials science and engineering. They are widely applied, such as optoelectronic devices, displays, lighting, biomarkers and sensors. The synthesis method of these materials by precipitation is highlighted as a simple and economical way to create nanoparticles, improving their properties. Therefore, it is understood that the synthesis of advanced ceramics, have great potential in photoluminescence applications, showing a high degree of technological innovation, discovering a significant advance and interest in the study area.

Verification of non-uniform strain fields in the presence of defects in isotropic and anisotropicmaterials via digital image correlation (DIC).

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High concentrations of defects can reduce a part performance, leading to its premature failure. In this way, the monitoring of defects or other types of imperfections in the materials, under deformation, contributes to the early identification of problems, preventing failures. The Digital Image Correlation (DIC) technique can provide both, the equivalent measures to conventional techniques and allow the visualization of the full-field deformation [1], which make it possible to identify defects by detecting irregular fields. A texture is applied over the reference image with spray paint and a grid is digitally created, which will be correlated in subsequent images using the correlation algorithm. This work aims to present a methodology for detecting defects through the evaluation of possible irregular strain fields and shear zones in composite specimens. Ten specimens were made according to the ASTM D-638 standard, with a polyester matrix and particulate reinforcement of red mud, which is an insoluble residue resulting from the dilution of bauxite alumina, in the Bayer process. The specimens were initially sanded and polished for subsequent texture generation by spray application, with white and black paint over the delimited area. The tensile test was carried out in the universal testing machine AROTEC WDW 100E, with a test speed of 1 mm/min. Images were captured using a CANON camera, model EOS REBEL T3i, 18 MP resolution, with a capture rate of one image per second. Preliminary results indicate that the DIC algorithm was able to generate the intended strain fields, as well as allowing to identify irregular fields that can be correlated to defects present in the material under test.

Acknowledgements:
Composites, defects, DIC.

References:

[1] M. Rodrigues et al. Determination of tensile properties of polyester - industrial waste red mud composites using digital image correlation (DIC). Materials Research, vol. 26, n. e20220449 (2023).