11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25
ICAM2009

Photo-reactive surfactant mediated synthesis of nanoparticles

André Galembeck[(1,2)]* and Rodrigo J. de Oliveira[(1)]

(1) Departamento de Química Fundamental, Universidade Federal de Pernambuco, CEP 50670-901, Recife – PE, BRAZIL, andre@ufpe.br
(2) Centro de Tecnologias Estratégicas do Nordeste - CETENE, Av. Prof Luiz Freire, 01, Cidade Universitária, Recife – PE, CEP 50740-540, BRAZIL
* Corresponding author.

Abstract – Surface active molecules which undergo photochemical reactions when irradiated with light can be labeled as photo-reactive surfactants (PRS). In this work, cobalt oxide nanoparticles were synthesized using reverse micelles formed by cobalt 2-ehyl-hexanoate, $Co(hex)_2$, in heptane as templates. The critical micelle concentration (cmc) was determined by viscosimetry. Formulations above cmc were submitted to UV-irradiation and the sample evolution was followed by UV-vis absorption spectroscopy (Figure 1) suggesting the formation of Co_3O_4, which was confirmed by high-resolution transmission electron microscopy (Figure 2).

Metallo-organic compounds such as 2-ethyl-hexanoate salts undergo thermal [1] or photochemical [2] decomposition which have been explored to synthesize oxide and metallic thin films or nanopowders. These molecules act also as surfactants, as we have recently confirmed by the formation of microemulsions in sodium 2-ethylhexanoate/heptane/water mixtures under proper conditions.

Surface active molecules which undergo photochemical reactions when irradiated with light can be labeled as photo-reactive surfactants (PRS). Such acronym is proposed in order to discriminate them from photo-sensitive surfactants (PSS), which suffer only conformational changes under light exposure. The photochemical decomposition of 2-ethyl-hexanoates result from ligand-to-metal charge transfer and gives CO_2, heptene and heptane as byproducts [2]. In this work, cobalt 2-ethylhexanoate ($Co(hex)_2$) was used as a PRS precursor to synthesize cobalt oxide nanoparticles (NPs) through a UV-induced photochemical reaction. $Co(hex)_2$ was synthesized by a hydrothermal route developed in our laboratory in which a cobalt salt and sodium 2-ethylhexanote are mixed in a sealed stainless steel reactor internally coated with teflon® and heated at 100ºC for 2 hours, leading to a blue solid that was dried under dynamic vacuum at room temperature.

Capillary viscosity of $Co(hex)_2$ solutions in n-heptane were measured at 30.00 ^{o}C. The viscosity increases as the $Co(hex)_2$ concentration is raised: near $2.0x10^{-3}$ mol·L^{-1} the slope is changed, suggesting the formation of reverse micelles [3]. A continuous change in the absorption spectra (and in the color sample, from blue to green) is observed when these $Co(hex)_2$/heptane formulations were irradiated with an UVC lamp (8W, 254 nm) as presented in Figure 1. The spectrum acquired after 7 hours of irradiation presents features of Co_3O_4: the transitions due to Co(III) and Co(II) sites in the spinel structure appear at 415 and 620 nm, respectively [4]. The cobalt oxide NP formation was confirmed by high-resolution transmission electron microscopy (HRTEM). The NPs average particle size is, nearly, 5 nm in diameter. Figure 2 shows a NP in which the (400) plans can be distinguished (d = 2.1 Å).

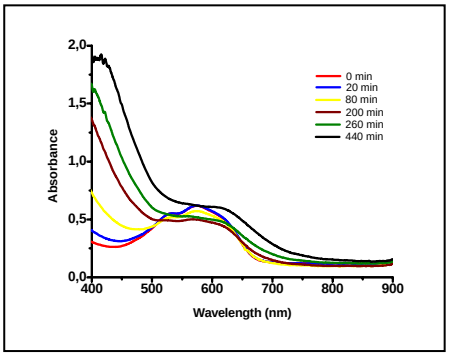

Figure 1: Absorption spectra of $Co(hex)_2$/heptane formulation submitted to UV-irradiation from 0 to 440 minutes.

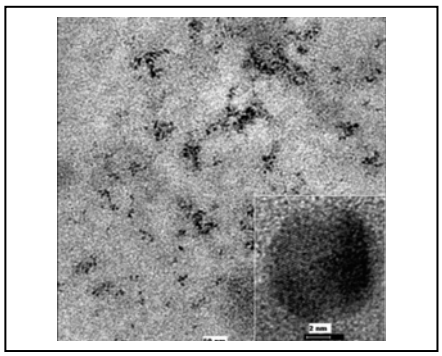

Figure 2: TEM image of Co_3O_4 nanoparticles. The inset is HRTEM of a single particle

[1] Y. Wang, I. Ramos, J. Santiago-Avilés, Nanotechnology 18 (2007) 295601.
[2] H.J. Zhu, R.H. Hill, Journal of Non-Crystalline Solids, 311 (2002) 174.
[3] K. Tyuzyo. Colloid and Polymer Science 175 (1961) 40.
[4] M. Ando, Kohei Kadono, Kenji Kamada, Koji Ohta, Thin Solid Films 446 (2004) 271.