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SiO_2/Nb_2O_5/graphite carbon ceramic conducting material: preparation, characterization, and its use as electrochemical sensor for 4-aminophenol

L. T. Arenas(1)*, T. C. Canevari(1) and Y. Gushikem(1)

(1) Instituto de Química, Universidade Estadual de Campinas, e-mail: leliz@iqm.unicamp.br
* Corresponding author.

Abstract – SiO_2/Nb_2O_5/graphite carbon ceramic (SiNbC) was prepared by the sol–gel processing method, this material was characterized by N_2 adsorption isotherms, scanning electronic microscopy coupled to energy dispersive spectroscopy and X-ray photoelectron spectroscopy. The results showed the Nb(V) homogeneously dispersed on the material surface. This material was used to investigate the detection of 4-aminophenol (4-AMP) by electrochemical methods. SiNbC presented good performance toward electrocatalytic oxidation of 4-AMP, this oxidation is mediated by the semiconducting property of Nb_2O_5.

Carbon ceramic electrodes (CCE) offer great prospects for electroanalytical applications due to their physical rigidity, porosity, easy modification and renewal surface[1,2]. In this work, SiNbC was synthesized and characterized. The sol-gel method used in the synthesis is based on the hydrolysis and policondensation of tetraethylorthosilicate (TEOS) and $NbCl_5$ in the presence of the carbon graphite particles in ethanolic solution, using HCl as catalyst. N_2 adsorption isotherms determined for this material were of type 1, typical of microporous materials, the surface area values (BET) is 267 m^2g^{-1} and pore volume is 0.067 cm^3g^{-1}. In the pore size distribution curve (BJH) is observed the presence of pores with diameter lower than 2 nm. From the SEM image and the corresponding EDS Si, C, and Nb mapping image (Figure1), it can be seen that all the elements are dispersed in the sub micrometric level, considering the magnification used. The XPS study revealed the existence bond of Nb-O in Nb_2O_5, Si-O in SiO_2, and C-C in graphite. The Nb3$d_{5/2}$ BE value is 208.7 eV, indicating an ionic character for Nb(V) species in the SiO_2 matrix, probably associated with Si–O–Nb linkages.

Figure 2a shows the cyclic voltammetric curve in presence of 4-AMP, where a defined redox couple at 0.27 V midpoint potential is observed. The absence of any redox process at the SiO_2/C-graphite electrode surface indicates that the 4-AMP oxidation is directly associated to the niobium oxide, a n-type semiconductor like some *d*-metal oxides. During the potential sweeping in the electrochemical investigation, 4-AMP compound can transfer electron to the conduction band producing the oxidized species. Figure 2b shows differential pulse voltammograms at different concentrations of 4-AMP, the anodic peaks increase proportionally with the concentration. The calibration curve plotted in the concentration range of 0.02–0.48 mmol l^{-1} resulted in a linear equation with a correlation coefficient of 0.993 and a detection limit of 0.95 µmol l^{-1}. The results obtained from this study lead us to conclude that SiNbC is a good candidate to be applied as electrochemical sensor for monitoring 4-AMP, considering its valuable properties such as low detection limit at reduced overpotential.

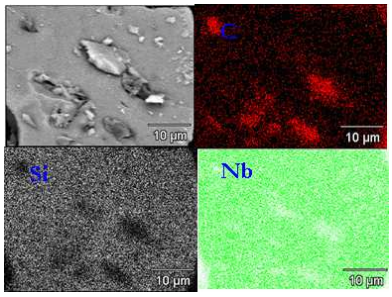

Figure 1: SEM image of SiNbC, and the corresponding energy-dispersive scanning (EDS) image of C, Si and Nb. The magnification was 1200x

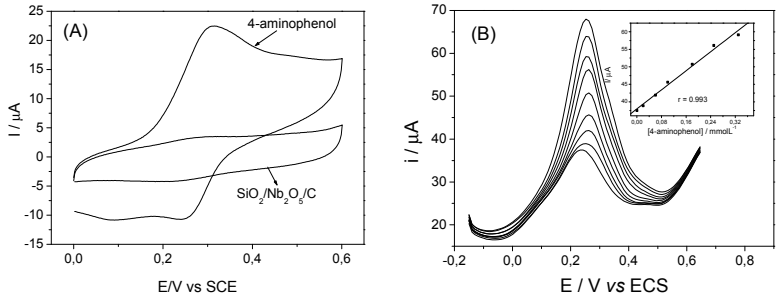

Figure 2: Cyclic voltammograms obtained with SiNbC in presence of 1mmol l^{-1} of 4-AMP (A). Differential pulse voltammograms for 4-AMP oxidation, at different concentrations. In 1 mol L^{-1} KCl pH 8. The inset figure shows the calibration curve.

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[2] Maroneze, C. M. Arenas, L. T. Luz, R. C. S. Benvenutti, E. V. Landres, R. Gushikem, Y. Electrochim. Acta. 53 (2008) 4167.