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Synthesis of Glassy Polymeric Carbon Modified with Metallic Ions

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Abstract – The production of glassy polymeric carbon (GPC) through the thermal treatment of a phenolic resin is described. The pyrolysis process has been investigated by Fourier-transform infrared spectroscopy and powder X-ray diffraction. The final material is a conductive non-porous glasslike material with rigid structure, which can be used as working electrode in electrochemical experiments.

Carbon based electrodes continue to be an intense focus of research, not only because of the diversity of applications which derive from their both electron donor/acceptor capacity and with the simultaneous presence of acidic/basic surface groups but also due to electrical properties, mechanical strength, easy processability and different forms attainable. The synthetic route to obtain glassy carbon is well known, as well as its structural and conductive properties, and its use in electrochemistry [1,2]. However, there is a class of based carbon material which possesses similar properties to glassy carbon. These materials, named glassy polymeric carbon (GPC), are produced from pyrolysis of phenolic or furfurylic resin, and they present thermal stability, robustness, mechanical strength, electrical conductivity and large potential range [2]. In this methodology, the resin – resol ($C_7H_8O_2$), in our case – is submitted to a thermal treatment at 1200 °C leading to GPC. In addition, before the heating, modifiers can be added to the resin changing mechanical, morphological, conducting, and electrochemical properties.

Thermal stability in oxidant atmosphere was verified by thermogravimetric analysis (Figure 1). The results indicate the carbon materials exhibit thermal stability up to 450 °C, *i.e.*, any reaction was observed and the materials preserved their initial properties. X-ray diffraction patterns of the pure GPC and the modified GPC, named GPC_{Fe} and GPC_{Cr}, depict peaks belonging to the glassy phase as well as diffraction peaks attributed to metal oxides, suggesting the production of a composite material (Figure 2). SEM images of the modified GPC surface (GPC_{Fe} and GPC_{Cr}) show cavities with 2 µm – 4 µm of diameter (Figure 3). Besides, it can be noticed that the both pure and modified GPC are consisted of lamellar arrangement of graphitic planes, as stated by Jenkins and Kawamura [1].

For modified GPC used as working electrodes, the cyclic voltammograms obtained in potassium ferricyanide aqueous solution show quite similar behavior in the presence of the $[Fe(CN)_6]^{3-}/[Fe(CN)_6]^{4-}$ redox pair obtained with pure GPC. Plots of I_p versus $v^{1/2}$ are straight lines evidencing that the mass transport is controlled by diffusion and that I_p is proportional to the concentration of electroactive species.

Overall, these results suggest that the GPC electrode presents satisfactory conductivity and allows electron transfer that is sufficiently rapid to approach reversibility. The agreement between the results obtained with pure GPC suggests that the former can be used in ordinary electrochemical experiments. Besides, the carbon materials exhibit suitable thermal stability for activation in oxidant atmosphere.

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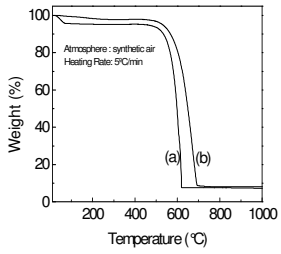

Figure 1: Thermogravimetric curves of: (a) GPC_{Ce} ; (b) GPC_{Fe}.

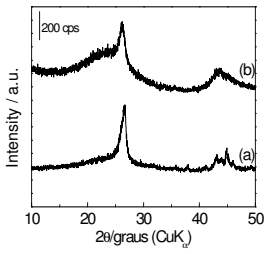

Figure 2: X-ray diffraction patterns of: (a) GPC_{Fe}, (b) GPC_{Cr}

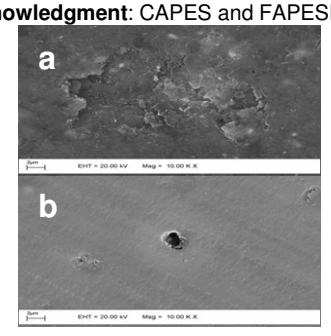

Figure 3: SEM images: (a) GPC_{Fe}, (b) GPC_{Cr}

References
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