Chemical shyntesis of graphene

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Introduction

Graphene is an individual two-dimensional, atomically thick sheet of graphite composed of a hexagonal network of sp^2-hybridized carbon atoms. It has been intensively investigated since 2004, when it was first isolatade.[1] It could be prepared by different routes, such as CVD (chemical vapor deposition), mechanical exfoliation, epitaxial growth of SiC, exfoliation of graphite oxide and chemical synthesis. However, obtaining graphene with defined size, shape and edge structure is still a great challenge to be overcome.[2] Chemical synthesis, even with some disadvantages, is considered the most promising for large-scale production, and allows a high control over the graphene structure. Aiming an alternative to solve problems related to chemical synthesis a new route of graphene synthesis was developed by the GQM-UFPR. This route has shown promise in the deposition on different substrates and the application of difficult materials to be processed.[3-4] This work aims to study variables of chemical synthesis of graphene from benzene in liquid-liquid interfaces to control every step of this process to improve the quality of the obtained graphene.

Results e Discussion

The samples have been obtained in a benzene/water system (10 mL of each) in which 2 g of anhydrous $FeCl_3$ was continuously added in portions of 100 mg. The system was kept under magnetic stirring for 3 h. After this time, the stirring was interrupted and a gray and continuous film was spontaneously obtained at the liquid interface. The film was deposited over different ordinary substrates and characterized by UV-VIS, Raman, SEM, EDS, X-ray and AFM. The variables studied were the time for addition of $FeCl_3$ (1, 2 and 5 minutes) and the cleaning of the films using the DCO method (sodium dithionite-oxalic acid-sodium carbonate).[5] This is a method for additional purification of the films due to the formation of iron oxides. DCO was used after cleaning with NH_4OH and H_2O.

The results obtained by Raman spectroscopy showed typical graphene profiles with graphene bands D, G and 2D. The UV-VIS spectra presented a π-π* transition at 265-270 nm. Graphene sheets of 500 nm^2 have been observed by SEM (Figure 1). In the sample without additional purification the presence of iron oxide can be observed (indicated by red arrow), confirmed by EDS spectrum (Figure 1C). The peaks at 2θ=33.3° and 2θ=36.0° observed in X-ray diffractograms (Figure 2) are characteristic of iron oxide. After cleaning by DCO method the iron oxide-based peaks decreased significantly and there was almost total reduction of the signal related to iron and oxygen in the EDS spectrum, showing thus its efficiency for removal of iron oxide present in the films. The sheets thicknesses obtained by AFM showed values between 5.39 and 8.60 nm.

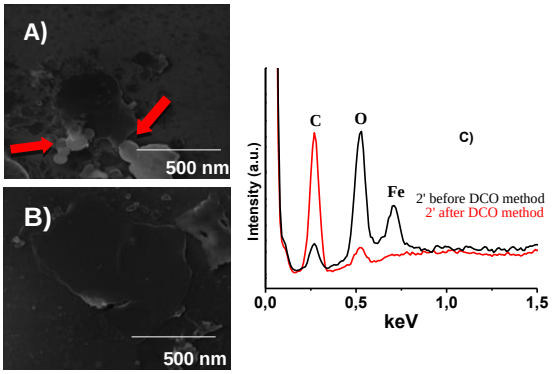

Figure 1. SEM images of samples: A) 2' before the cleaning by DCO method, B) 2' after the cleaning by DCO method, C) EDS spectrum.

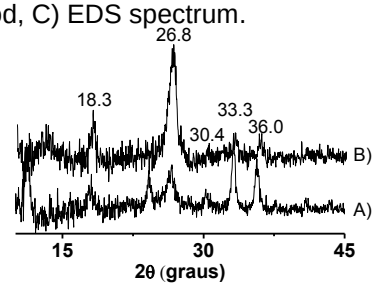

Figure 2. X-Ray diffractograms A) 2' before the cleaning by DCO method and B) 2' after the cleaning by DCO method).

Conclusions

The interfacial route is a new, innovative, versatile and very efficient way to chemically-obtained graphene. The purity of the samples can be controlled by a DCO method, in which iron oxide obtained as side product is efficiently removed.

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