

Synthesis and characterization of graphene synthesized from graphene oxide by the modified polyol process

Rebecca F. Albers^{1*}(PG), Laudemir C. Varanda¹(PQ).

¹Grupo de Materiais Coloidais, Instituto de Química de São Carlos - Universidade de São Paulo.

*refalbers@gmail.com

Key words: graphene, polyol process, graphene oxide.

Introduction

Graphene, a two-dimensional, single-layer sheet of sp^2 hybridized carbon atoms,¹ has attracted tremendous attention of the scientific community due to its exceptional physical, thermal and electric properties. The most employed method to prepare graphene sheets is the chemical oxidation of graphite to obtain graphene oxide (GO),² followed by exfoliation/reduction to achieve graphene. A variety of reduction methods has been applied, but the most of them require high energy cost and long reaction time to yield the product. In this work, the polyol process³ was used like a new alternative route to produce graphene and graphene/nanoparticles composites in a single step.

Results and Discussion

GO was prepared by the conventional modified Hummers method² and characterized by infrared spectroscopy (FTIR), Figure 1. The spectrum revealed the characteristic organic functions of the material: stretching O-H (3390 cm^{-1}), C=O (1740 cm^{-1}), C=C (1630 cm^{-1}) and C-O (1230 cm^{-1}).

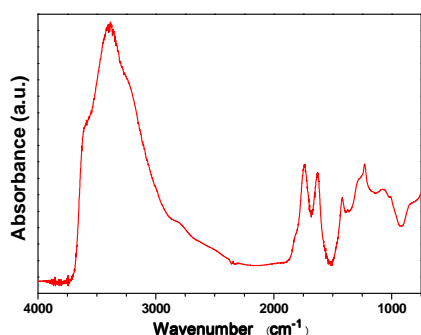
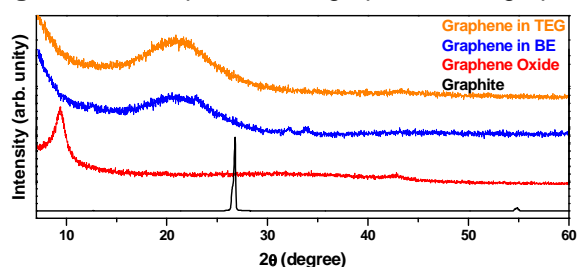


Figure 1. Infrared spectrum of GO.

Comparing the X-ray diffraction (XRD) patterns of the precursor graphite (Sigma Aldrich) and the synthesized GO (Figure 2) it is possible to infer the increasing distance between the carbon atoms planes. Calculated spacing (d) using the Bragg's law resulted in values of $d = 3,3\text{Å}$ and $d = 9,4\text{Å}$ for graphite and GO, respectively. The GO was suspended in ethylether and this suspension was used in the polyol process to synthesize graphene sheets. The polyol process composition was the same used to synthesize nanoparticles in the GO absence, such as: surfactant (oleylamine), reducing

agent (tetradecanediol) and a high boiling point solvent. Two different solvents are evaluated: benzyl ether (BE) and tetraethylene glycol (TEG) and using both it was obtained graphene samples, which showed a diffraction halo in 20° (2θ), indicating the graphene formation.

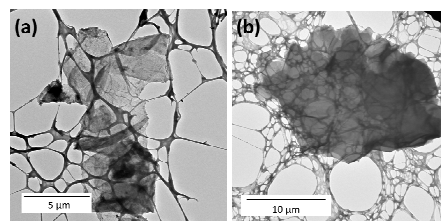
Figure 2. XRD patterns of graphite, GO, graphene



using BE and TEG as solvent.

Graphene sheets were analyzed by TEM, Figure 3, comprising the quality of the sheets obtained in both solvents.

Figure 3. TEM images of graphene synthesized in



(a) BE and (b) TEG. Scale bar: 5 (a) and 10 (b) μm .

Conclusion

The polyol process showed a good strategy to obtain graphene from GO. This new synthetic route to exfoliate and reducing GO is faster and cheaper than other reported. Also, it is an alternative to produce composite graphene/nanoparticles in a single step, according to the experimental conditions, which will be tested presently.

Acknowledgments

To FAPESP.

¹Geim, A. K.; Novoselov, K. S. The rise of graphene. *Nat. Mater* 2007, 6 (3), 183-191.

²Marcano, D. C.; Kosynkin, D. V.; Berlin, J. M.; Sinitskii, A.; Sun, Z.; Slesarev, A.; Alemany, L. B.; Lu, W.; Tour, J. M. Improved Synthesis of Graphene Oxide. *ACS Nano* 2010, 4 (8), 4806-4814.

³Varanda, L. C.; Jafelicci, M. Self-Assembled FePt Nanocrystals with Large Coercivity: Reduction of the fcc-to-L10 Ordering Temperature. *J. Am. Chem. Soc.* 2006, 128 (34), 11062-11066.