

An Electrochemical Method for the Production of Graphite Oxide

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We report on the development of a technique for the rapid production of exfoliated graphite oxide, (GO) using an electrochemical oxidation and exfoliation method. A vitreous carbon foam sheet was utilised as the anodic material in conjunction with a steel bar cathode. The anode was oxidised under an applied electrical field of 30V and a current of 2A in a 1M NaOH 0.6 M KCl 200ml electrolyte solution. The post regime GO product, collected after evaporation of aqueous electrolyte, was analysed using scanning electron microscopy (SEM), atomic force microscopy (AFM), energy-dispersive X-ray spectroscopy (EDAX) and ultraviolet-visible spectroscopy. The preliminary results show that the solution was found to contain nanoscale GO flakes with an average length of 5nm.

The Hummers' method is the *de facto* method of choice for the synthesis of graphite oxide (GO), however the process involves the use of a dangerous chemical cocktail of strong oxidising agents that require cooling to avoid spontaneous ignition [1-3]. This reported method for GO production finds its advantage in the fact that the use of hazardous materials is almost completely avoided. The second advantage of the electrochemical technique is that it produces graphite oxide flakes in useable quantities within hours, not days. Comparable electrochemical methods for producing GO have utilised graphite rods as the electrode materials [4, 5]. As such the production of GO has taken up to 7 days to reach similar sized nanoflakes [4] or used current densities of 600A/m² and temperatures of 600°C [5]. However, the use of vitreous carbon foam provides a far higher surface area for the oxidation reactions to occur and thus accelerating the process [6]. A third advantage is that the GO produced is potentially purer than that produced using the Hummers method, due to the fewer chemicals used in the synthesis. These three advantages combined significantly increase the ease at which GO can be produced for applications of large scale i.e. for large surface area coating. As such, the

supply of GO for any future graphene based technology fabricated on an industrial scale can be readily satisfied.



Fig. 1. The vitreous carbon foam anode, supported in a PTFE frame, showing the loss of carbon material which has been converted to GO.

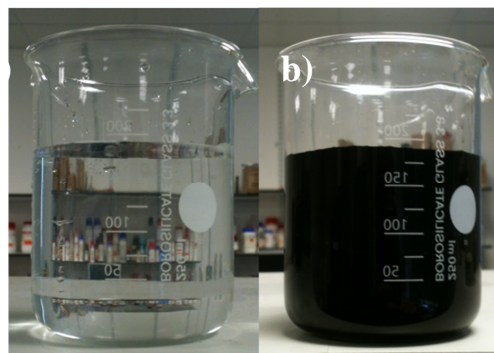


Fig. 2. The electrolyte solution before a) and after b) application of the electric field after two hours.

- [1] W. S. Hummers, R. E. Offman, J. Am. Chem. Soc., 80, 1958, p1339
- [2] S. Stankovich, D. A. Dickin, Carbon, 45, 2007, p1558
- [3] H. A. Becerril, J. Mao, R. M. Stoltenberg, Z. Bao, Y. Chen, J. Am. Chem. Soc., 2, 2008, p463.
- [4] A. -L. Cui, G. -X. Feng, Y. -F. Zhao, H. -Z. Kou, H. Li, G. -H. Zhu, H. -S. Hwang, H. -C. Oh, Y. -J. Kwon, D. -C. Lee, Electrochem Commun, 11, 2009, p409.
- [5] A. Zhamu, J. Jang, B. Z. Jang, US Patent Application Publication, No. US 2009/0026086 A1, Jan, 2009.
- [6] C. E. Hunt, Y. Wang, App. Surf. Sci., 251, 1-4, 2005, p159.