# Production of High-Quality Exfoliated Graphene using a Two-Step Electrochemical Approach

#### Edward P.L. Robertsa

Ashutosh K. Singha, Maria Perez - pageb, Nael Yasria, and Stuart M. Holmesb

<sup>a</sup> Department of Chemical & Petroleum Engineering, University of Calgary, 2500 University Dr NW, Calgary, Canada; b Department of Chemical Engineering and Analytical Science, University of Manchester, Manchester UK

edward.roberts@ucalgary.ca

### **Abstract**

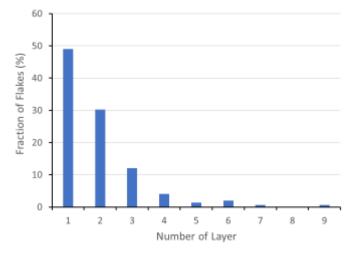
Conventional chemical exfoliation, such as Hummer's method, provides a cost-effective pathway for large-scale production, however, the method suffers from several key challenges such as the usage of corrosive and toxic reagents, high processing temperatures, long duration, the requirement for chemical reduction of the produced graphene oxide, and the poor quality of graphene produced which severely affects its potential applications. Recently, electrochemical exfoliation of graphene has been proposed for a high production yield with better quality. Two step electrochemical exfoliation involves: (1) intercalation of ionic species between the graphene layers, followed by (2) oxidation of these ionic species under an anodic voltage into gas molecule enabling exfoliation of graphene flakes from the graphite electrode [1].

In this study we examine a new approach to the intercalation, using an intercalating ion that has received very little attention and has not been evaluated for electrochemical exfoliation. The intercalation was followed by exfoliation in (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution leading to the production of graphene flakes. This approach enhances the rate of exfoliation, and reduces the amount of oxidation experienced by the graphene layers during exfoliation. The number of layers in the particles of electrochemically exfoliated graphene (EEG) was determined by Atomic Force Microscopy. It was found that >90% of the EEG flakes were three layers of graphene of fewer, with 50% single layer and 30% two layer graphene (See Figure 1. Compared with other methods, the as produced EEG was found to have a less disrupted sp2 lattice structure, with a high yield of about 95% of graphene, along with high electrical conductivity and a low oxidation level (with a C:O ratio of about 15, determined by XPS).

## References

[1] J. Cao, P. He, M.A. Mohammed, X. Zhao, R.J. Young, B. Derby, I.A. Kinloch, R.A.W. Dryfe, J. Am. Chem. Soc., 139 (2017), 17446-17456.

# **Figures**



**Figure 1:** Histogram of the number of graphene layers in the as-produced EEG flakes , obtained by AFM.