Synthesis of Few Layer Graphene by Electrochemical Intercalation and Exfoliation Induced by a Polymeric Surfactant

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Graphene, one-atom-thick planar sheet of sp^2 -bonded carbon atoms, is a quasi-2-dimensional (2D) material. The fascinating properties of graphene and few layer graphene (FLG) have made it one of the most promising materials.

However, graphene suffers from a problem that is common to many novel materials, the lack of a method for producing it in large quantities. So, fully exploiting the properties of graphene requires yet the development of a method for their mass production. The exfoliation of expandable graphite via graphite intercalation compounds (GICs) can be one of the most attractive methods for this proposes because this process, which has been studied considerably in the past is cheep and scalable.2 The electrochemical intercalation and subsequent exfoliation of graphite is a well known phenomenon in the field of lithium-ion batteries using carbonaceous material.

Here we report a simple method to produce graphene that combines the anodic and cathodic electrochemical intercalation/expansion of graphite in aqueous perchloric acid and posterior expansion by microwave radiation and exfoliation with ultrasound radiation (Figure 1).

The morphological characterization of the EG shows the typical worm-like shape of the EG since our method rely in the intercalation/expansion principle.

Raman results suggest that the material integrity is retained, and the pre-treatment has no negative influence on the material integrity. The level of exfoliation and the structure of the material dispersed have been analyzed by TEM and Raman, confirming that few-layer graphene have been successfully produced (Figure 2).

Graphene is usually dispersed in organic solvents such as NMP and o-DCB. Here we reported the dispersion of few layer graphene in water with the aid of a polymeric surfactant. The polymer is a PVA modified with perylene groups. The aromatic planar molecules ensure interaction with graphene while the polar polymer confers to the system solubility in water.³

The absorption spectrum of PVAPery/Grf dispersions confirms the presence of graphene laminates. In addition, upon exposure to a laser beam, the Tyndal scattering effect is observed for the PVAPery/Grf dispersions (Figure 3).

The presence of dispersed graphene laminates was further confirmed by Raman and NIR spectroscopy as well as with fluorescence measurements.

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Figures

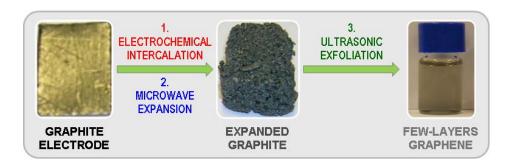


Figure 1. Schematic representation of the process to produce few layer graphene from a graphite electrode

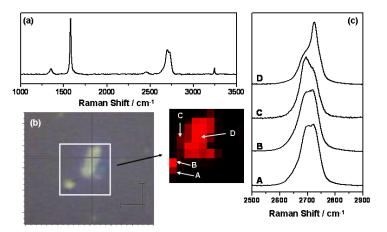


Figure 2. (a) Raman spectrum observed from exfoliated -1V FLG sample deposited on a quartz slide, (b) optical image of a typical deposit on the quartz slide and false-color map of the zone indicated, and (c) representative spectra from the points indicated of the 2D region of FLG.



Figure 3. The Tyndal scattering in a solution of PVA-Perylene containing dispersed few layer graphene