Controlled Synthesis of Large and Small Graphene Oxide Sheets

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Abstract

Graphene oxide (GO) sheets have been synthesized using a modified-Hummers method previously described in [1]. The GO materials are well dispersed with brownish color and stable at room temperature for more than 6 months. The yield of the reaction is between 7 and 11% with a final concentration between 1 and 2 mg/mL in water at neutral pH*.* The GO production has been successfully up-scaled under safe conditions and with an accurate controlled temperature set-up. The lateral dimension of the GO sheets has been controlled by drying the large GO material (< 20µm, Figure 1A), using freeze-drying and oven incubation, and then sonicating for 5 min to obtain the small GO sheets (< 400nm, Figure 1B) without introducing changes in the surface properties. The structural properties such as lateral dimension and thickness of the materials have been studied by Atomic Force Microscopy (AFM), optical microscopy and Dynamic Light Scattering (DLS). Optical properties have been studied by using UV-Vis and spectrofluorimetry. Surface properties of the GO material have also been studied by Raman spectroscopy and ζ–potential. The Raman spectrum shows D and G bands at 1319cm^{-1} and 1596cm^{-1}, respectively, characteristics of most poly-aromatic hydrocarbons. The D to G band intensity ratio (I_D/I_G) calculated, corresponding to the metric of disorder in the graphitic structure, is 1.4. And the surface charge measured with a Zetasizer instrument shows an average zeta-potential of -50 mV, highly negatively charged as expected for GO materials. To elucidate the functionalization degree, thermo-gravimetric analysis (TGA) indicated that a total of 40% of the GO material has been functionalized. X-Ray photoelectron spectroscopy (XPS) has been used to quantify the purity of the GO (>99.5%), C:O ratio (~2.3, C 70% : O 30%) and the contribution of each individual functional group such as carboxylic, carbonyl, epoxides and hydroxyls. We show that large (< 20µm) and small (< 400nm) GO flakes can be controllably synthesized in a reproducible and up-scalable manner.

References

[1] Ali-Boucetta H*, et al.* Adv Healthc Mater., **2**(3) (2013) 433-441.

Figures

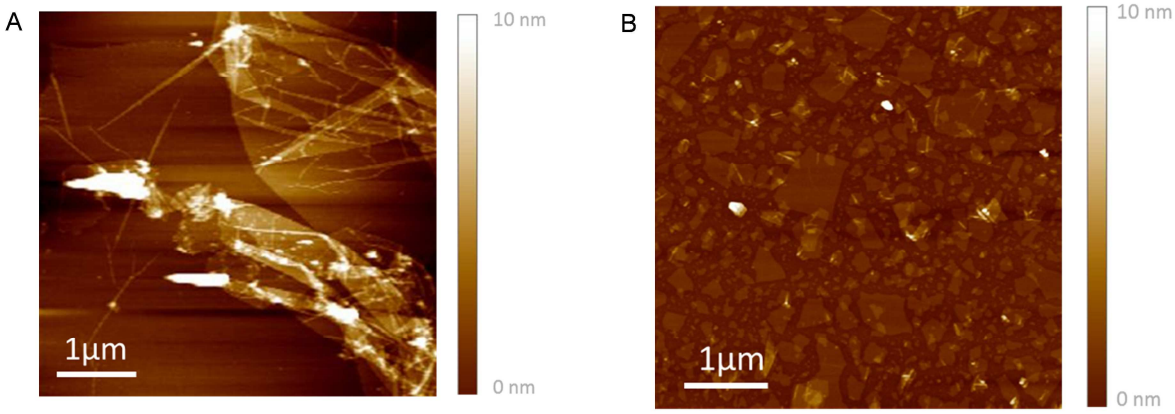

Figure 1. AFM height images for **(A)** large GO; and **(B)** small GO.