

Supplementary Information

Facile fabrication of few-layer graphene and graphite nanosheets by high pressure homogenization

Jingqi Shang, Feng Xue, Enyong Ding*

College of Material Science and Engineering, South China University of Technology, 381 Wushan Road, Guangzhou 510641, PR China.

* Corresponding author. Tel.: +86 020 87111290. E-mail address: eyding@scut.edu.cn

Experimental and Characterization

In a typical experiment, all the chemical reagents were of analytical grade and obtained commercially, and 500 mg graphite power (300 meshes, Aladdin) was dispersed in 200mL DMF in a sealed bottle at an initial concentration of 2.5 mg/ml. The mixtures were subjected from 70MPa to 100MPa using a high pressure homogenizer (AH-1000D, ATS) for 10 min at 25°C through an external cooling apparatus, and then centrifuged for 60 min at 5000 rpm to remove larger flakes. The supernatant was collected by pipette carefully, after that, the few layer graphene were obtained. In the similar way, we homogenized the graphite in pure water under the pressure of 100MPa for 10 min, and then filtered the solution, and the upper solid was the graphite nanosheets. We compute the concentration of the graphene dispersion by heat weighting method. In a typical process, 3mL graphene was dropped in the weighting bottle and then dried at 80°C in the vacuum drying oven to evaporate the solvent. In order to calculate the yield of graphene, the amount of un-exfoliated or thick graphite flakes was measured quantitatively by vacuum filtration.

The following characterizations of graphene were the sample under the pressure of 100MPa. UV-Vis absorption spectroscopy of graphene was performed with a UNICAN UV-500 instrument for the range

from 190 to 800 nm using 1cm quartz. Raman spectroscopy was performed by LabRAM Aramis with He-Ne laser (633 nm) as the irradiation light source. The samples were prepared on a Si/SiO₂ wafer with an oxide layer of 300 nm. Atomic force microscopy (AFM) studies were carried out in a tapping mode on a NanoScope III (R) instrument after depositing a drop of the diluted dispersions on freshly cleaved mica and drying in the ambient condition. The surface morphology of the samples (graphene and graphite nanosheets) were observed by scanning electron microscope (SEM) and field scanning electron microscopy (FE-SEM, LEO 1530 VP). Prior to the SEM analysis, all the samples were platinum-coated using a sputter coater. Transmission electron microscope (TEM) (FEI-Tecnai 12) and high resolution TEM (HRTEM) (JEM-2010HR) were operated with an accelerating voltage of 200 KV and a micro-grid copper covered with carbon film was used to deposit the graphite. The absorption coefficient of graphene dispersion was computed by UV-Vis absorption spectroscopy (Fig. S1). The graphene dispersion was diluted to 0.00065, 0.013, 0.0026, 0.0052, 0.0065, 0.0078 mg/mL respectively. The absorbance (660 nm) divided by cell length was plotted versus concentration, follows the Lambert-Beer law with an average absorption coefficient of $\alpha_{660\text{nm}}=3463 \text{ mL}/(\text{mg}\cdot\text{m})$ which is a little difference from other reference. The graphene concentration was about 0.084mg/mL and the yield was about 2.68%. The concentration of our graphene dispersion is slightly higher than most pure solvents without any surfactants, but it is relatively much lower comparing with the methods of adding the surfactants or exfoliating in the ionic liquid.

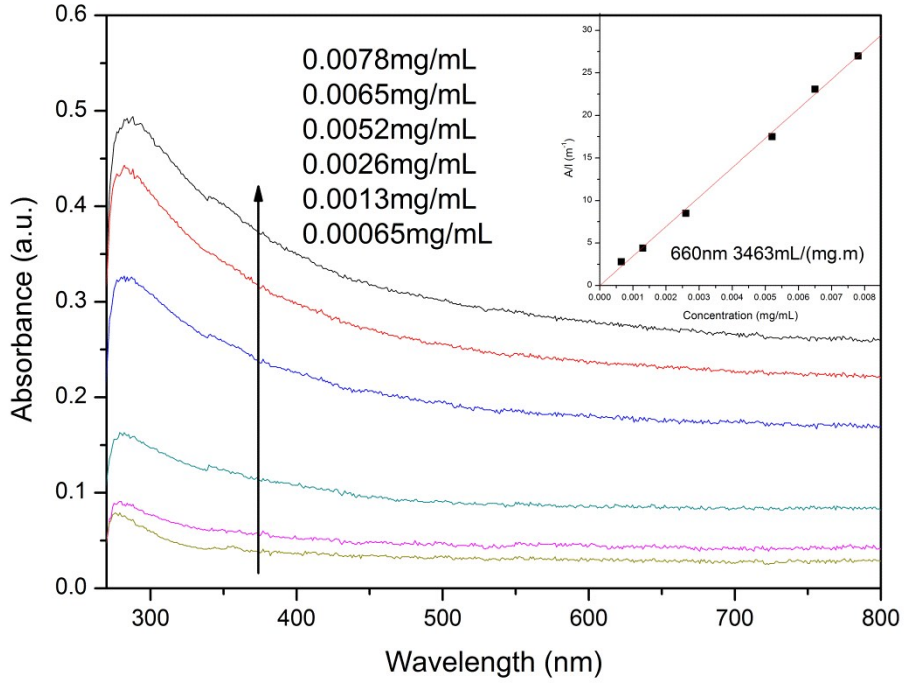


Fig. S1 UV-Vis absorption spectra of the diluted graphene dispersion. The six different spectra represent the concentrations from bottom to top, which stand for 0.00065, 0.013, 0.0026, 0.0052, 0.0065, 0.0078 mg/mL. Inset: optical absorbance divided by the cell length as a function of different concentrations. The solution follows the Lambert–Beer law with an average absorption coefficient of $\alpha_{660nm}=3464 \text{ mL}/(\text{mg.m})$

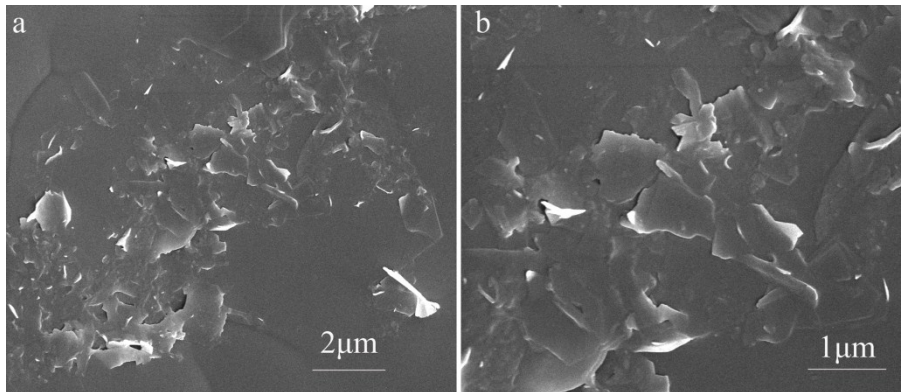


Fig. S2 FE-SEM images of graphene

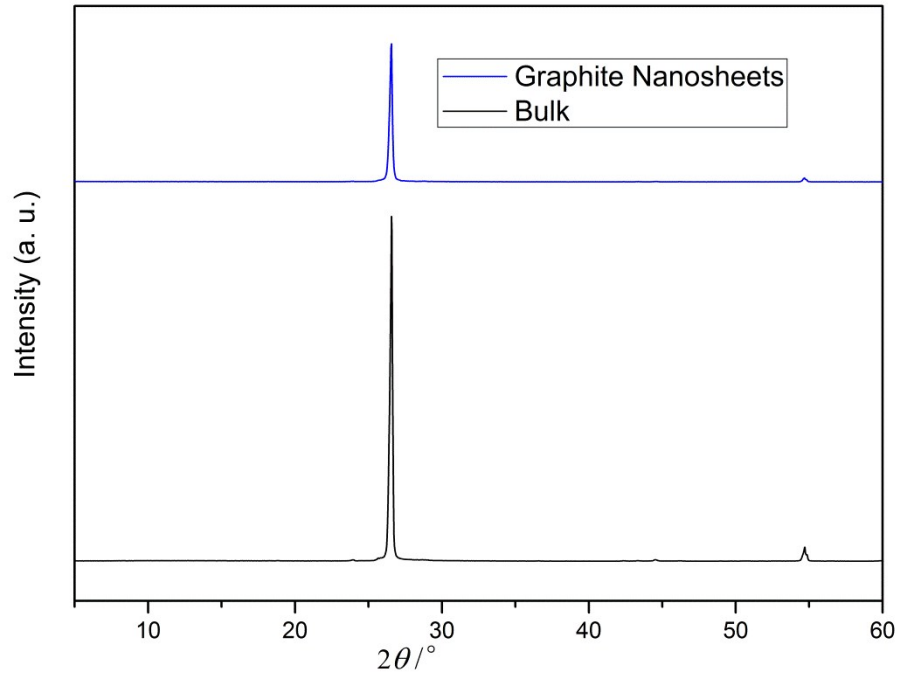


Fig. S3 XRD for graphite bulk and exfoliated graphite nanosheets

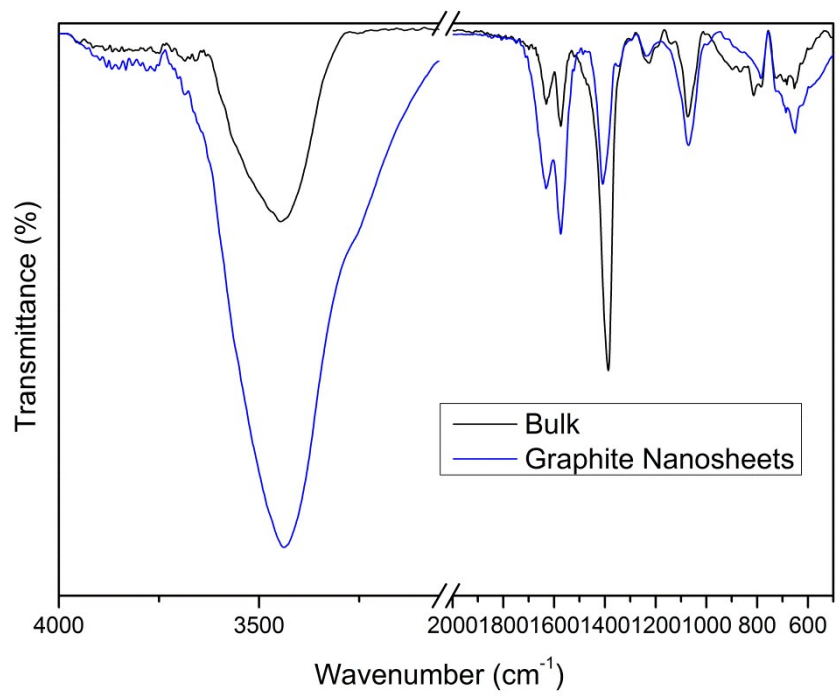


Fig. S4 FT-IR for graphite bulk and exfoliated graphite nanosheets