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Electronic Supplementary Information

Effective Production of Nano-sized Graphene via Straight-forward Exfoliation of Microcrystalline Graphite

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Experimental Section

In a typical experiment, 0.1 g microcrystalline graphite (mc-G) flakes (chenzhou, Hunan province) were dispersed homogeneously in 40 ml N-methyl-2-pyrrolidone (NMP, content > 98%, Guoyao) through a low power ultrasonic bath using a KQ-100DE Ultrasonic Cleaner for 10 min. Then the pre-dispersed mc-G dispersions were ball milled at 40 Hz for 12 h on a planetary mill (QM-3SP2, Nanjing University Instrument Company). During the ball-milling the weight ratio of three sized zirconia balls (diameter of 5, 2, 1 mm, respectively) were kept at 1:3:1. After that, centrifugation was performed at 4000 rpm for 30 min using a Cence Centrifuge H1850 to precipitate the un-exfoliated graphite flakes and the impurities. Finally, the top 80% of the dispersions were collected and characterized.

Transmission electron microscopy (TEM, JEM-2010 JEOL) and high-resolution transmission electron microscopy (HRTEM, HITACHI, H-7650) were performed to investigate the morphology of the product nano-sized graphene (NG). Atomic force microscopy (AFM) images were collected on a Nanoscopy Multimode IIIa scanning probe microscopy system (Veeco Instruments Ltd) in tapping mode to determine accurately the thickness of the graphene sheets. Raman spectroscopy was recorded with a Lab RAM Aramis spectrometer (laser wavelength 532 nm). X-ray photoelectron spectroscopy (XPS) analyses were carried out on a VG Escalab MK II spectrometer (Scientific Ltd). X-ray diffraction measurements were performed on X'Pert Pro with Ni-filtered Cu Kα radiation. UV-Vis absorption spectra were taken using a UV-1600 spectrophotometer (Beijing Rayleigh Analytical Instruments) with quartz cuvettes (10 mm optical path). Photoluminescent spectra were measured at room temperature using a F4500 spectrophotometer (Hitachi Ltd).

Determination of absorption coefficient: The nano-sized graphene flakes (0.1 g) were dispersed in N-methyl-2-pyrrolidone (40 ml) through a low power ultrasonic bath for 10 min. After that, centrifugation was performed at 4000 rpm for 30 min to recover the dispersions. Then the above dispersions were divided into two fractions with a same known volume. The NO.1 dispersions were filtered through poly-vinylidene fluoride filters (PVDF, pore size of 0.22 μm) to directly weigh the amount of dispersed graphene and determined the actual graphene concentration. The NO.2 dispersions were analyzed by UV-1600 spectrophotometer. Several dilutions were done from known concentration graphene dispersions. Then the absorbance at a wavelength of 660 nm was measured to determine the absorption coefficient.

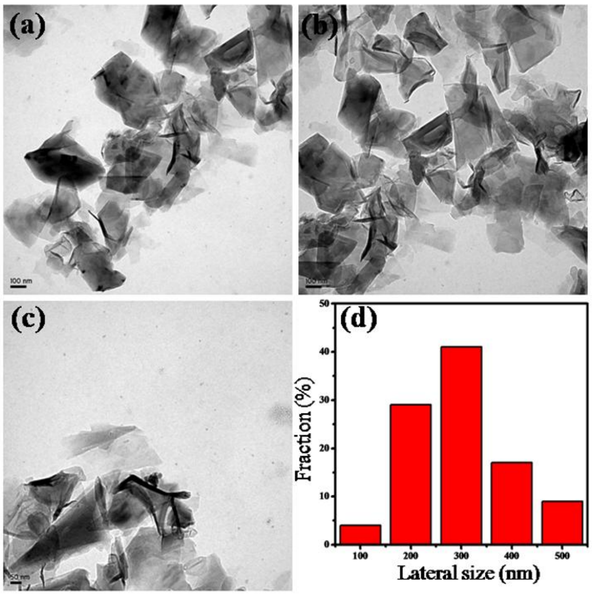
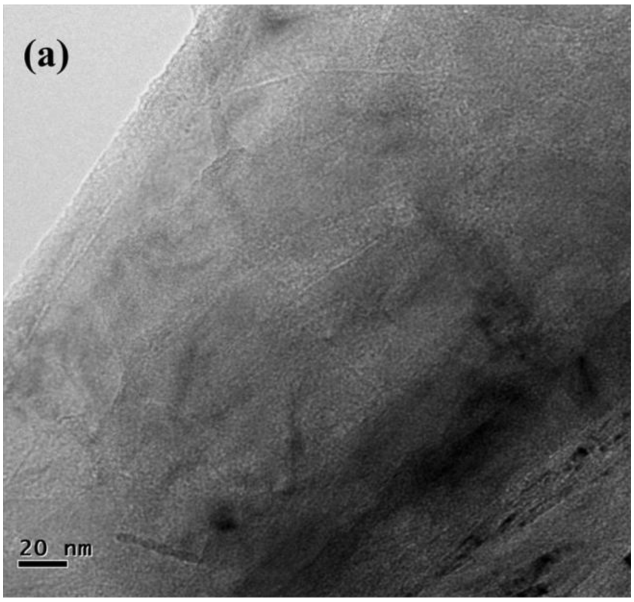


Fig. S1 (a), (b), (c) TEM images of mc-G sheets deposited onto grids.

(d) Statistical flakes size analysis of mc-G sheets by TEM.



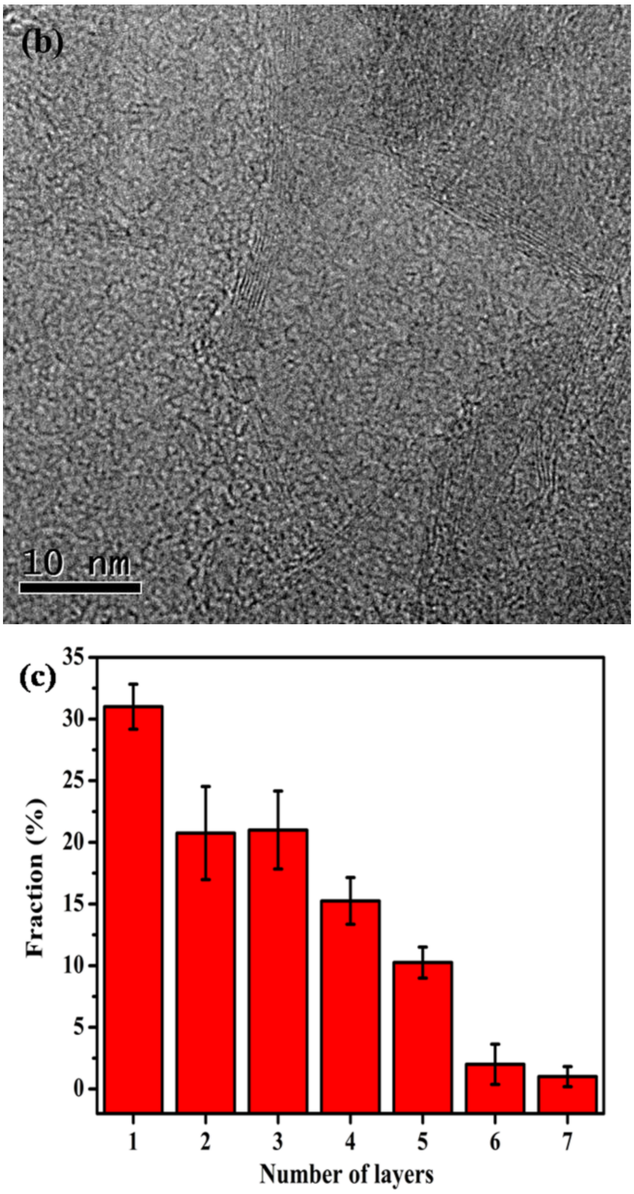


Fig. S2 (a), (b) Representative HRTEM images of NG sheets to verify the exfoliation.

(c) Statistical flakes thickness analysis of NG sheets by HRTEM.

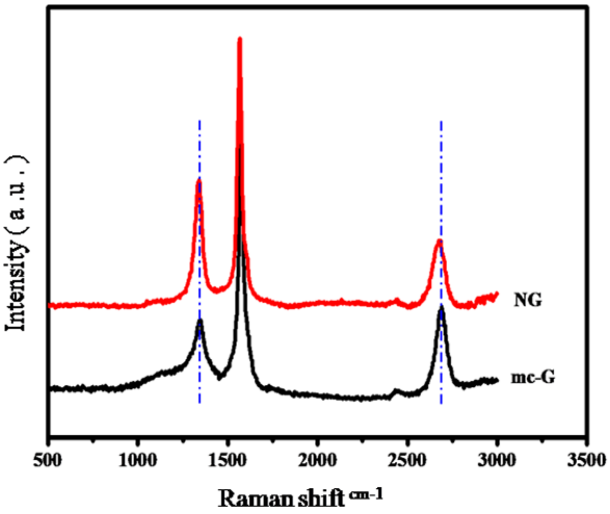


Fig. S3 Raman spectra of mc-G and NG

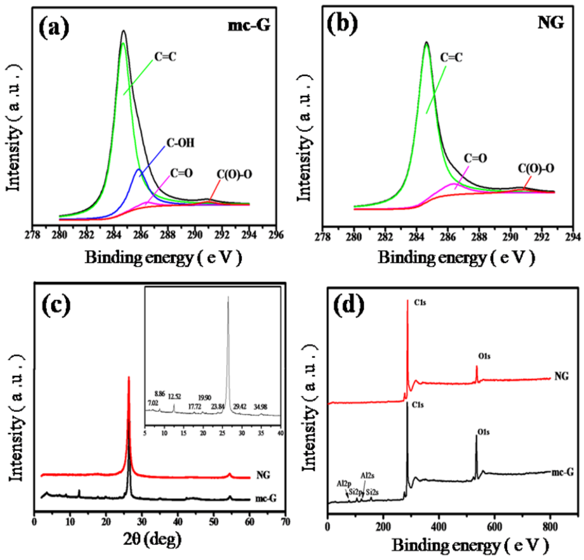


Fig. S4 (a) XPS C 1s spectra of mc-G. (b) XPS C 1s spectra of NG.

(c) XRD spectra of mc-G and NG. (d) XPS full scan spectra of mc-G and NG