Electronic Supplementary Information

High-yield, large-scale production of few-layer graphene flakes within seconds: using chlorosulfonic acid and H_2O_2 as exfoliating agents

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Experimental Section

Chlorosulfonic acid (CSA) was purchased from Zhejiang Juhua Co., Ltd. (Zhejiang, China). All the other chemicals were purchased from Aladin Ltd. (Shanghai, China) and used as received without further purification. The water used throughout all experiments was purified through a Millipore system.

Graphene flakes were prepared as follows: In a typical experiment, 50 mg of graphite powder was added in 6 mL of CSA solution (99.9 wt %) under a gently shaking. Then, 3 mL of H_2O_2 (30 wt %) was added to the solution dropwise under stirring. The products were harvested by centrifugation, followed by washing three times with distilled water to remove the excess acids. After that, the products were dried under vacuum and then re-dispersed in CSA. The dispersion was centrifuged at 4,000 r.p.m. for 15 min to remove the unexploited and partial exfoliated graphite.

Atomic force microscopy (AFM) was conducted with a SPI3800N microscope (Seiko Instruments, Inc.). X-ray photoelectron spectroscopy (XPS) analysis was measured on an ESCALABMK X-ray photoelectron spectrometer. The graphene flakes were examined by X-ray diffraction (XRD) using a Rigaku D/Max-2550 diffractometer with Cu Ka radiation ($\lambda = 1.54056$ Å) at 50 kV and 200 mA in the range of 10–60° (2θ) at a scanning rate of 5° min^{-1}. Raman spectra were obtained on J-Y T64000 Raman spectrometer with 514 nm wavelength incident laser light. Scanning electron microscopy (SEM) measurements were made on a XL30 ESEM FEG scanning electron microscope at an accelerating applied potential of 20 kV. The sample for SEM characterization was prepared by placing a drop of the dispersion on a bare indium tin oxide coated glass substrate (ITO) and air-dried at room temperature. Transmission electron microscopy (TEM) measurements were made on a HITACHI H-8100 electron microscopy (Hitachi, Tokyo, Japan) with an accelerating voltage of 200 kV.

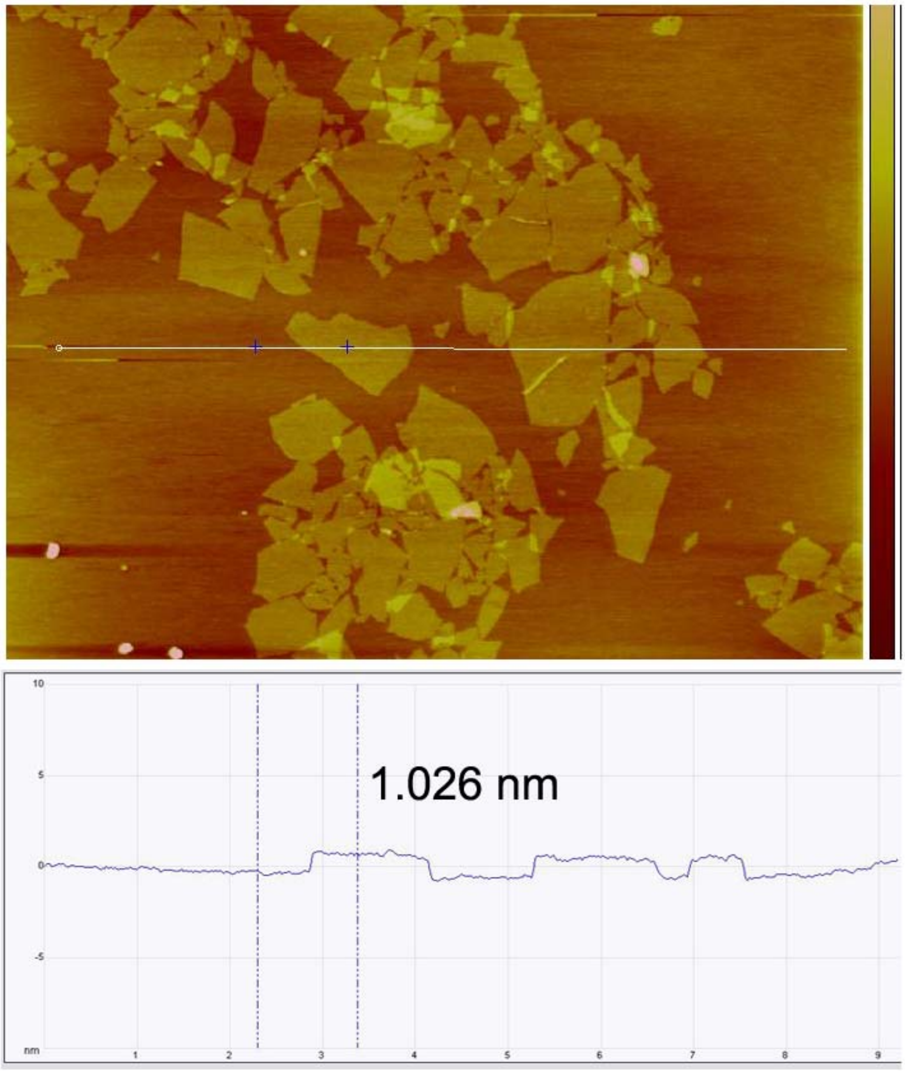

Fig. S1 AFM analysis on the graphene flakes.

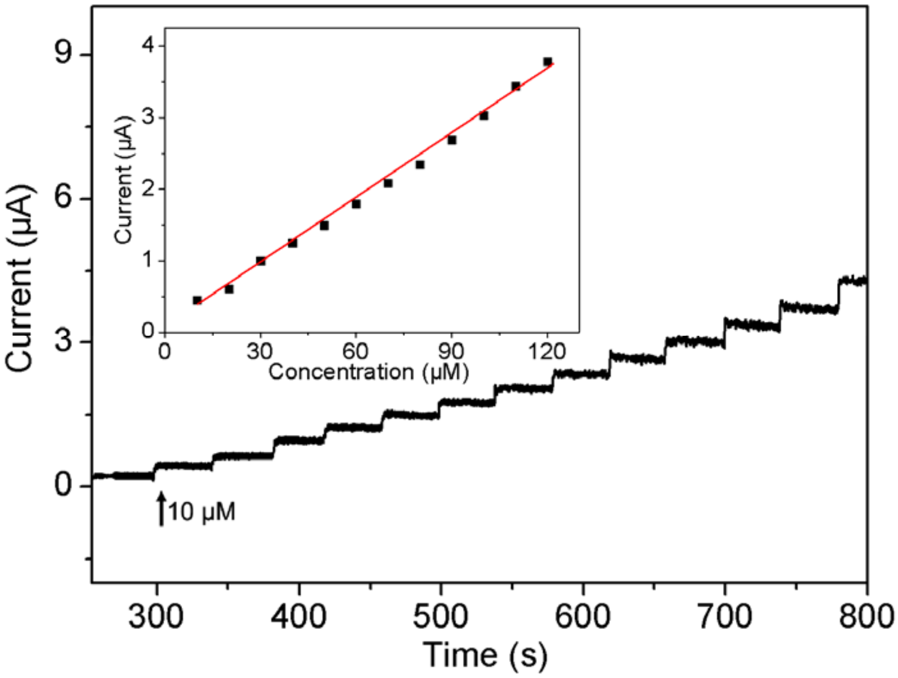

Fig. S2 Typical steady-state response of the G/GCE to successive injection of AA solution into the stirred 0.2 M PBS at pH: 7.4 (Applied potential: 0.1 V).