

Electronic Supplementary Information (ESI) for:

One-pot Reduction of Graphene Oxide at Subzero Temperatures

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1. Supporting figures

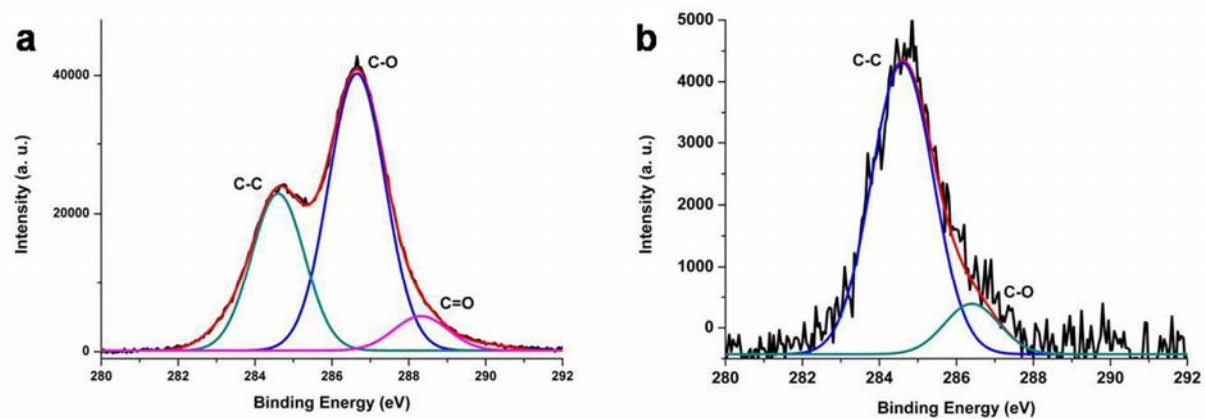


Fig. S1. XPS spectra of (a) GO and (b) rGO. After chemical reduction, all related oxygen peaks decreased.

2. Experimental details.

2.1 Materials

Natural graphite (Bay Carbon, SP-1 graphite), Sulfuric acid (95-97%), hydrogen peroxide (30 wt%), potassium permanganate, sodium nitrate, hydriodic acid (57 wt%), trifluoroacetic acid and sodium bicarbonate were obtained from commercial sources and used without further purification.

2.2 Synthesis of GO.

GO was prepared from natural graphite powder (Bay Carbon, SP-1 graphite) using a modified Hummer's and Offenman's method with H_2SO_4 , NaNO_3 , and KMnO_4 .¹

2.3 Synthesis of rGO powder.

In a three-necked round flask, 4.0 g GO was dispersed in 1 L of trifluoroacetic acid with sonication and hydriodic acid (80 mL) was added. This reaction mixture was stored at $-10\text{ }^\circ\text{C}$ for 40 h with stirring. Filtration was used to purify the product. The product was washed with saturated sodium bicarbonate, deionized water and acetone, and dried overnight in vacuum at room temperature.

2.4 Characterization.

Elemental analysis was conducted with an LECO 932 Elementary Analyzer (Atlantic Microlab, Inc.). HR-TEM imaging of rGO was performed using a JEOL JEM-2100F microscope at 400 KeV. The powder XRD pattern was acquired using a D8-Advance instrument (Germany) and $\text{Cu-K}\alpha$ radiation. All XPS measurements were taken by a SIGMA PROBE (Thermo VG) using a monochromatic $\text{Al-K}\alpha$ X-ray source at 100W. Raman spectroscopy measurements were performed on a micro-Raman system (Renishaw, RM1000-In Via) with an excitation energy of 2.41 eV (514nm). The thermal properties of the rGO were characterized by TGA (Polymer Laboratories, TGA 1000 plus).

3. Dynamic process of GO powder reduction by HI/TFA

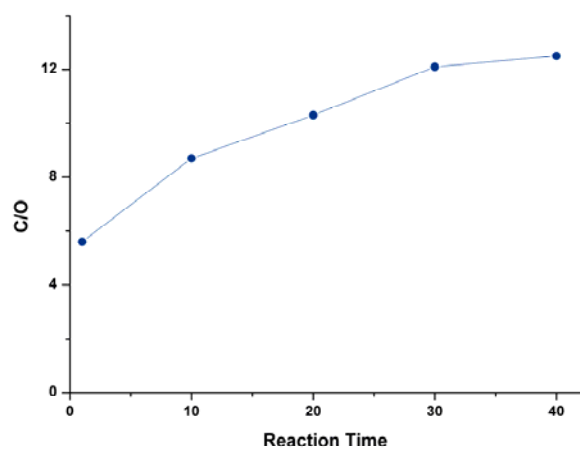


Fig. S2 Variation of the C/O atomic ratio of rGO powders with reaction time (hours) as a result of TFA/HI reduction at -10 celsius degree.

Referemces

1. (a) Hummers, W. S.; Offeman, R. E. *J. Am. Chem. Soc.* **1958**, *80*, 1339-1339. (b) Cote, L. J.; Kim, F.; Huang, *J. Am. Chem. Soc.* **2008**, *131*, 1043-1049.