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More about the mechanism



Figure S1: The possible fluorination mechanism in Carbon, 2012, 50, 5403-5410.

Experimental details

Materials

Graphite flakes (>99.99%, 325 mesh, Alfa Asea), HNO₃ (~65%), HF (~47%) and all other chemicals are of analytic grade and were used directly as received without any further purification.

Synthesis of graphene oxide

GO was prepared following a modified Hummers method. Graphite flake (2.0 g) and NaNO₃ (1.0 g) were added to concentrated H_2SO_4 (46 mL) under vigorous stirring in an ice bath. Then KMnO₄ (9.0 g) was gradually added while the temperature of the suspension was kept below 20 °C. Successively, the reaction system was warmed to a 40 °C and vigorously stirred for 2 h. Then, 92 mL water was added slowly producing a large exotherm to 98 °C, and the reaction system was maintained at 98 °C for 30 min with external heating introduced. Then 280 mL water and 20 mL H_2O_2 (30%) was added, and the color of the solution turned to yellow from dark brown. After cooling down to room temperature, the mixture was filtered and washed with HCl (5%) three times and washed with water until sulfate could not be detected. This was then dispersed in water under ultrasonic and centrifuged at 9000 rp/min for 5 min. The decanted supernatant was dried to obtain the graphene oxide solid.

Synthesis of fluorinated graphene oxide

50 mg GO was dispersed in 40 mL ultrapure water under ultrasonic, then centrifuged to remove any insoluble substances. The supernatant was transferred into a 50 mL Teflon-lined stainless steel autoclave, then 5 mL concentrated HNO₃ and 5 mL HF was added while gently stirring. The autoclave was heated at 180 °C for 12 hours and naturally cooled to room temperature. The resultant solution was directly evaporated to dryness in water bath and the solids as prepared are fluorinated graphene oxide denoted as FGO. Control sample denoted as NGO was synthesized following the same process except that 5 mL ultrapure water was substituted for 5 mL concentrated HNO₃.

Materials characterization

The structure of FGO was characterized with transmission electron microscope (TEM, JEM-2100F). X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi, ThermoFisher-VG Scientific) was performed with a monochromated Al-Kα X-ray source. The Fourier-transform infrared (FTIR, ET 6700, Thermo Scientific) spectrum was measured by pressing potassium bromide troche. GO, NGO and FGO were dispersed in ultrapure water at a concentration of 2mg mL⁻¹ for ultraviolet-visible (UV-Vis) absorption spectrometry measurements on UV-1801 spectrophotometer (Beifen-Ruili). The GO dispersion was diluted due to its dark colour.