Electronic Supplementary Material (ESI) for ChemComm.

High-yield electro-oxidative preparation of graphene oxide

1 Experiments:

1.1 GO synthesis by electrochemical exfoliation:

In a typical synthesis experiments, natural graphite was connected to the anode and platinum wire served as the cathode. A 0.2 M buffer solution of sodium citrate (pH 3.7) was used as the electrolyte. The electrochemical experiments were conducted at 300 mA constant current for 5 hours using PSS-210-GW INSTEK programmable power supply equipped with Instek PSU software. After the electrolysis, the suspension produced was taken from the cell and centrifuged at 5000 rpm for 20 min to remove the large non-exfoliated particles.

1.2 Characterization of the produced GO:

Samples for Raman spectra the Atomic Force Microscope (AFM), and SEM analysis were prepared by a simple drop casting method. The suspension resulting from the electrochemical process was diluted in distilled water with 1:20 suspension to water volume ratio and then deposited on 300 nm thick SiO_2 covered on Si wafers. The layers were subsequently dried at 80 ^oC. The sample for the TEM analysis was deposited from the diluted suspension on TEM grids and dried overnight at 80 ^oC.

Raman spectra were obtained using a Renishaw system 1000 spectrometer coupled to a He-Ne laser. The laser spot size was ~1-2 μm, and the power was about 1 mW when the laser is focused on the sample using an Olympus BH-1 microscope. Atomic force microscope (AFM) images were obtained using a Multimode Nanoscope V scanning probe microscopy (SPM) system (Veeco, USA) with Picoscan v5.3.3 software. Tapping mode was used to obtain the images under ambient conditions. The morphology of the graphite and GO were also observed by SEM using a Carl Zeiss SUPRA SMT AG scanning electron microscope (LEO1525, Carl Zeiss, Oberkochen, Germany) with the accelerating voltage at 5kV. The TEM analysis used a FEI Tecnai F20 microscope.

Samples for the XRD and thermogravimetric analysis (TGA) was obtained by filtration of the suspension using Anodisc® alumina membranes with 100nm pore size. The XRD analysis was conducted using a Philips X'PERT APD powder X–ray diffractometer (λ = 1.54 Å, CuK_α radiation). TGA was performed in air and under argon atmosphere

were carried out using a Jupiter Netzsch STA 449 C instrument. The sample was placed into alumina crucible and heated with a rate of 10°C/min from 30°C up to 800°C. To avoid thermal expansion of the GO due to rapid heating, GO samples were also heated from 50°C to 500°C at 1°C/min.

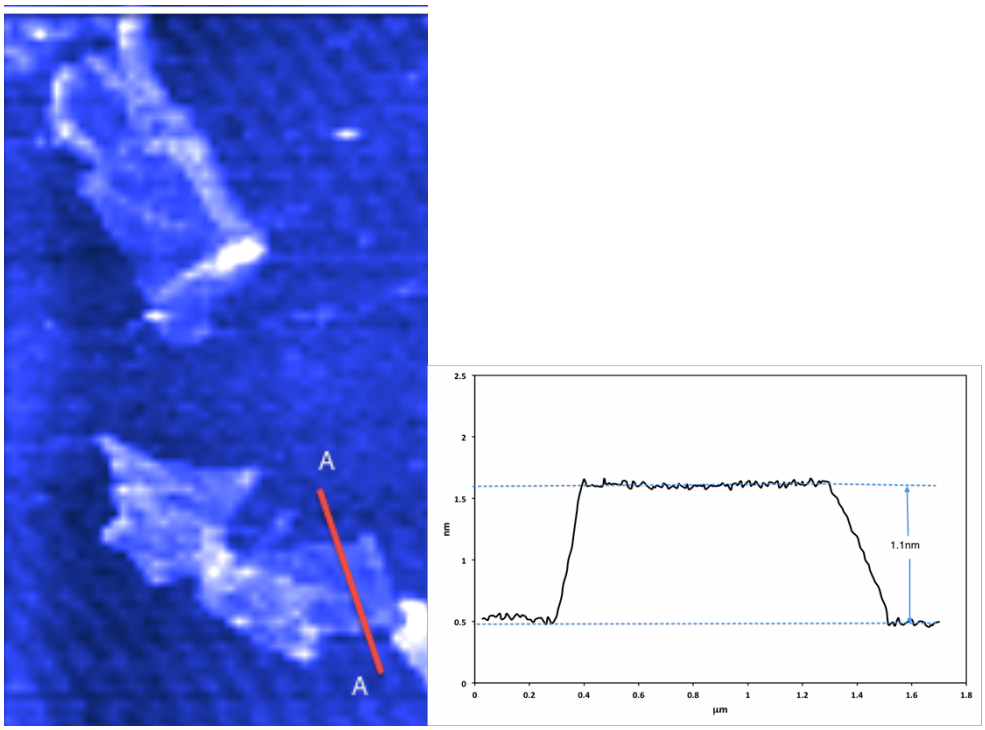

Figure S!: AFM images for typical monolayer and the height profile

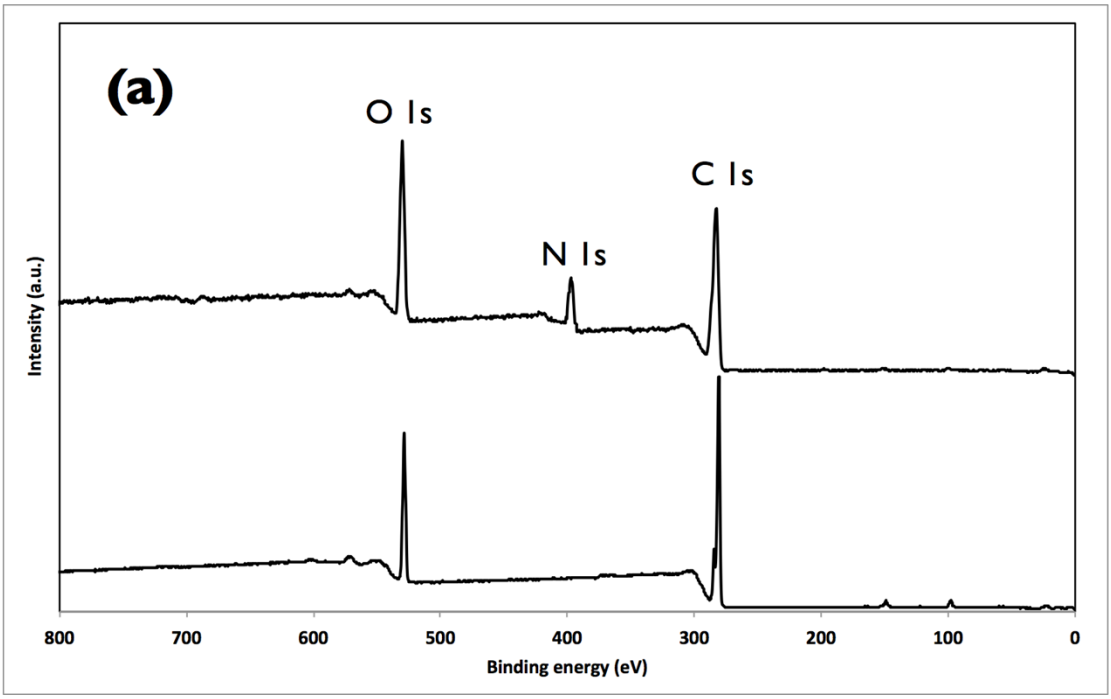

Figure 6: XPS spectra for the produced GO from buffer solution(bottom) and when nitric acid was added to the electrolyte (top)

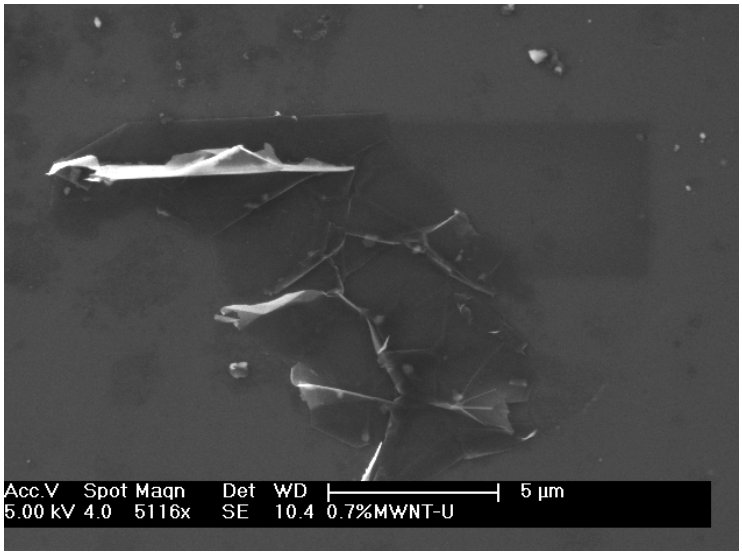

Figure 7: SEM image of GO produced from puffer solution after 5 hours of electrolysis

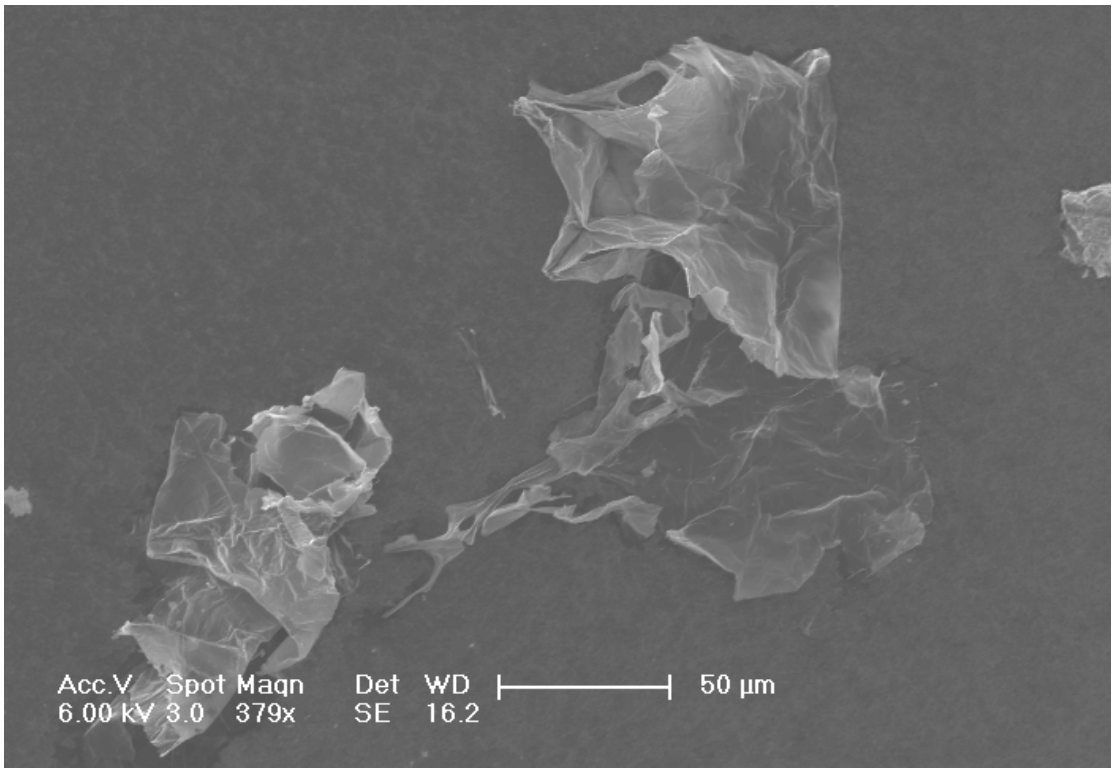

Figure 8: SEM image of t the produced GO when HNO3 was added to the electrolyte.