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Effective Electron Transfer between Heteropoly Blue and Graphene Oxide: A Green Approach to Graphene Synthesis

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Preparation

$K_{12.5}Na_{1.5}[NaP_5W_{30}O_{110}]$ (NaP_5W_{30}):

In a typical experiment, 33 g of $Na_2WO_4{\cdot}2H_2O$ was dissolved in 30mL of water and 26.5 mL of 85% H_3PO_4 was added. The mixture was placed in a Teflon-lined autoclave and was heated at 120 °C for 12 h. After the autoclave was cooled to room temperature, 15 mL of water and 10 g of KCl solid was added to the resultant yellow solution resulting in the formation of precipitate. The precipitate was filtered off and washed with 2 mol/L KAc and methanol. Then the collections were re-dissolved in 30 mL of hot water (90 °C). When the hot solution was cooled to room temperature, white crystals formed. A second recrystallization gave the pure product.

Graphene oxide (GO):

Graphene oxide (GO) was synthesized following Hummers method by reacting natural graphite powder in a mixture of concentrated H_2SO_4, $NaNO_3$ and $KMnO_4$. After the reaction finished, H_2O_2 was added to the reaction vessel. The GO solution was seperated by centrifugation and washed twice with 1M HCl and ten-times with water. The products were dried in vacuum to give brown sheets. GO aqueous solution was prepared by ultrasonically dissolving a required amount of GO solid into deionized water.

Characterization

UV-vis spectra were measured on a Beckman DU 800 Spectrophotometer. X-ray diffraction (XRD) patterns were obtained using a Bruker D8 ADVANCE X-ray diffractometer with Cu Kα radiation λ=1.5418Å. Atomic force microscopy (AFM) images were recorded on a Agilent 5400 in tapping mode. X-ray photoelectron spectroscopy (XPS) measurements were performed on a Thermo ESCALAB 250 spectrometer with a monochromic X-ray source (Al Kα, 1486.6 eV) and the charging shift was corrected by the binding energy of C1s at 284.6eV. Raman spectra were obtained with a LabRAM HR UV-NIR Raman Microscope (λ=532nm). All electrochemical experiments were carried out on a Model 660D Voltammetric Analyzer (CH Instruments, USA) in a conventional one-compartment cell with a glassy carbon electrode (GCE) with a diameter of 3mm or a Indium tin oxide (ITO) electrode with a surface area of 1cm×2cm as working electrode, a Ag/AgCl as reference electrode and a Pt wire as

auxiliary electrode. Prior to electrochemical modification, a GCE was successively polished with 0.3 and 0.05 mm α-Al_2O_3, and ultrasonically washed water between each experiment and dried for 5 min. ITO was cleaned firstly with acetone twice by ultrasonication and then washed with amounts of deionized water for 10min. All measurements were carried out at room temperature under nitrogen atmosphere.

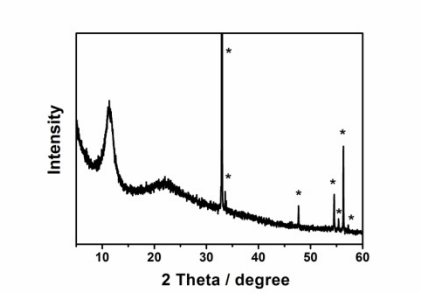

Fig.S1 XRD patterns of reduced GO immobilized on Si slide. Diffraction peaks represented by star signals origins from Si substrate.

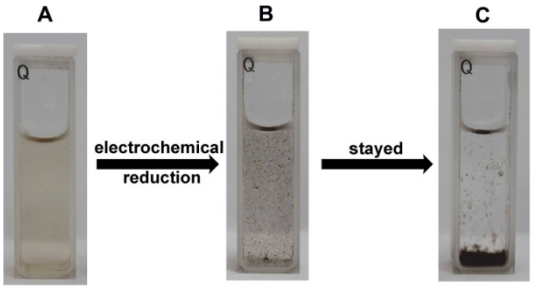

Fig.S2 Photographs of a 0.5 mmol/L NaP_5W_{30} and 0.05 mg/ml GO aqueous solution containing 5 mmol/L HCl and 20 mmol/L NaCl (A), the solution scanned by two hundred CV cycles (B), the solution laid overnight (C).

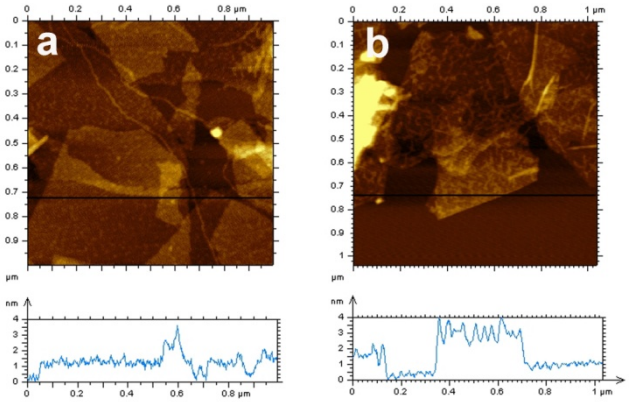

Fig.S3 AFM images of GO (a) and RGO (b) deposited on freshly cleaved mica substrates with height profiles below.

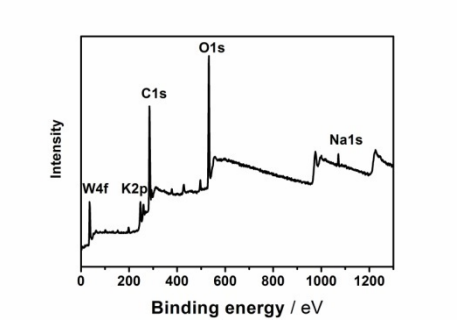

Fig.S4 XPS spectrum of RGO showing the presence of W and K atom.

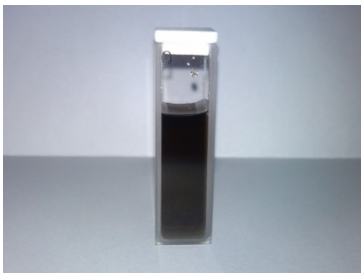

Fig.S5 Photo of 0.5 mg/mL RGO aqueous solution.

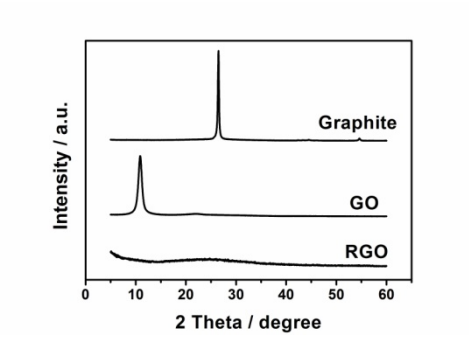

Fig.S6 XRD patterns of graphite, GO and RGO.