

Supporting Information for
SYNTHESIS OF COVALENTLY BONDED GRAPHENE OXIDE–IRON MAGNETIC
NANOPARTICLES AND ITS KINETICS OF MERCURY REMOVAL

Paul N. Diagboya,^{*, a, b} Bamidele I. Olu-Owolabi,^b Kayode O. Adebowale^b

^a *National Center for Nanoscience and Technology, Beijing 100190, China*

^b *Department of Chemistry, University of Ibadan, Ibadan, Nigeria*

*Corresponding author: Tel: +234 80 3805 2553. E-mail: pauldn2@yahoo.com

S1: Preparation of GO and synthesis of GO-FMNP hybrid

GO was prepared by chemical exfoliation of natural flake graphite, followed by repeated purification cycles using centrifugation. The centrifugation steps are 5 000 rpm (at 5 min) twice, 8 000 rpm (at 5 min) once, 10 000 rpm (at 5 min) once, 10 000 rpm (at 10 min) once, and 11 000 rpm (at 30 min) once.

Typically, 10 g of natural graphite (99 wt% purity; average particle diameter of 20 μm; Yingshida Graphite Co. Ltd., Qingdao, China) and 7.5 g of NaNO₃ were added into a 2 L beaker. 600 mL of H₂SO₄ was added to the beaker and a magnetic stirrer chip. The mixture was stirred while cooling in an ice water bath. 40 g KMnO₄ was added gradually over a one hour period. The cooling was continued for about 3 h, and the mixture was allowed to stand for five days at about 20 °C with gentle stirring. 1 L of 5 % vol/vol H₂SO₄ aqueous solution was added over a 1 h stirring period, and stirring continued for 2 h. This was followed by addition of 30 mL H₂O₂ and further stirring for 2 h. This was followed by the centrifugation steps described above. After each centrifugation step, Millipore ultra-pure water was used to make up the volume in the centrifugation tubes.

The concentration of the exfoliated graphene oxide after centrifugation was determined by weighing 10g of the GO dispersion in a pre-weighed round bottom flask and drying to constant weight. The weight of the dry GO was used to calculate the concentration of the GO dispersion in mg/mL.

Table S1: Synthesis of GO-MNP

	APS-FMNP	GOMNP-0	GOMNP	GOMNP-1	GOMNP-2
	MNP-APS	GO-MNP _{no EDC; no APS}	GO-MNP _{plus APS; no EDC}	GO-MNP _{0.5}	GO-MNP _{1.0}
MNP (mg)	0.3	0.3	0.3	0.3	0.3
Ethanol (mL)	20	20	20	20	20
Sonicate (min)	10	10	10	10	10
APS (mL)	3	–	3	3	3
Incubation	60°C; 4hr	60°C; 4hr	60°C; 4hr	60°C; 4hr	60°C; 4hr
100 mM EDC (mL)	–	–	–	4	4
‘x’ mg/mL GO conc.	–	1.0 mg/mL	1.0 mg/mL	0.5 mg/mL	1.0 mg/mL
Sonicate (min)	10	10	10	10	10
Incubation	65°C; 8hr	65°C; 8hr	65°C; 8hr	65°C; 8hr	65°C; 8hr

‘x’ is the final solution GO concentration.

S2: Descriptions of instruments and methods

IR spectra have been obtained using a Spectrum-1 FTIR spectrometer (Perkin–Elmer Instruments Co. Ltd, USA) in the scanning frequency of 4500 – 450cm⁻¹. Thermo-gravimetric analysis (TGA) was carried out on the various powdery samples using (TGA) using Pyris Diamond Thermogravimetric/differential thermal analyzer (PerkinElmer Instruments Co. Ltd., USA) by heating the samples at 5 °C min⁻¹ to 800 °C in nitrogen atmosphere. The Raman spectra of pristine GO and the GP samples were observed using a Renishaw inVia Raman spectrometer (Renishaw plc, UK). All samples were tested in powder form on silicon wafer without using any solvent. The laser excitation was provided by a regular model laser operating at 514 nm. XRD patterns of the samples were measured from 3.0° to 90° by a Philips X’Pert PRO X-ray diffraction instrument (PANalytical B.V., Netherlands). The surface area and porosity measured using a Micromeritics ASAP 2020 M + C accelerated surface area and porosimetry analyzer (Micromeritics Instrument Corporation, USA). The samples were degassed at 80 °C and the nitrogen adsorption–desorption isotherms obtained were evaluated to give the

Brunauer–Emmett–Teller (BET) specific surface area and pore volume. 1000 mg/L stock solution of Hg^{2+} was prepared by using Millipore water and 0.01 M NaNO_3 as indifferent electrolyte. 50 mg/L solution of Hg^{2+} was prepared from this when needed. Hg^{2+} adsorptions were monitored using the Varian 710-ES ICP optical emission spectrometer.

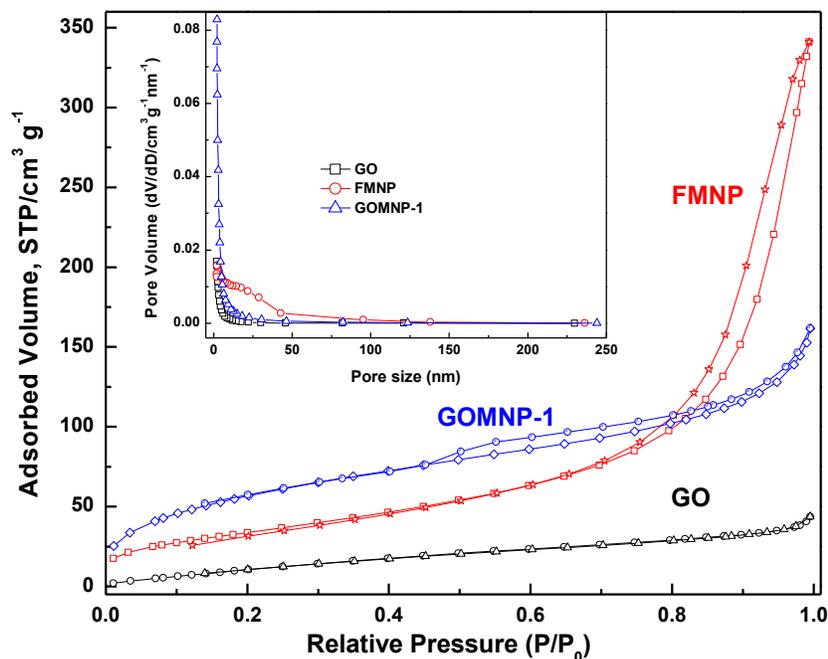


Figure S1: BET surface areas (A) and porosities (B) of GO, FMNP and GOMNP-1 showing surface areas of 56, 125 and 214 m²/g, respectively; and adsorption average pore diameters of 4.8, 13.7, and 13.1 nm, respectively. The unbroken and broken curves depict the nitrogen adsorption and desorption, respectively.

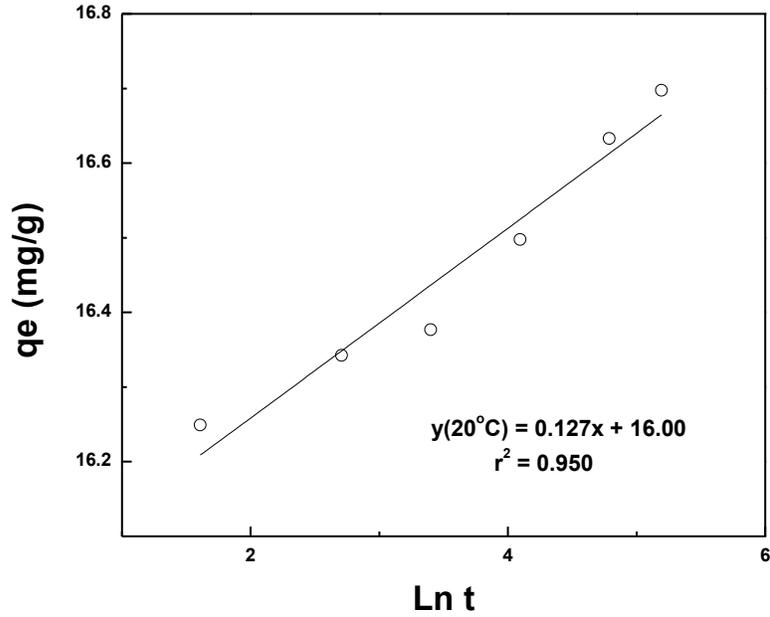


Figure S2a: Elovich kinetics model isotherm at 20 °C (plot of $\log qt$ vs $\ln t$)

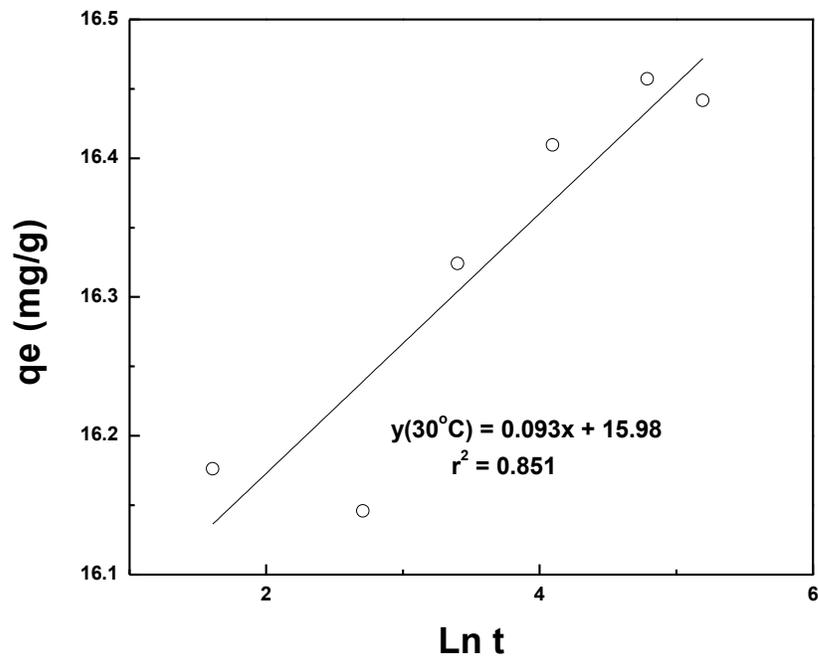


Figure S2b: Elovich kinetics model isotherm at 30 °C (plot of $\log qt$ vs $\ln t$)

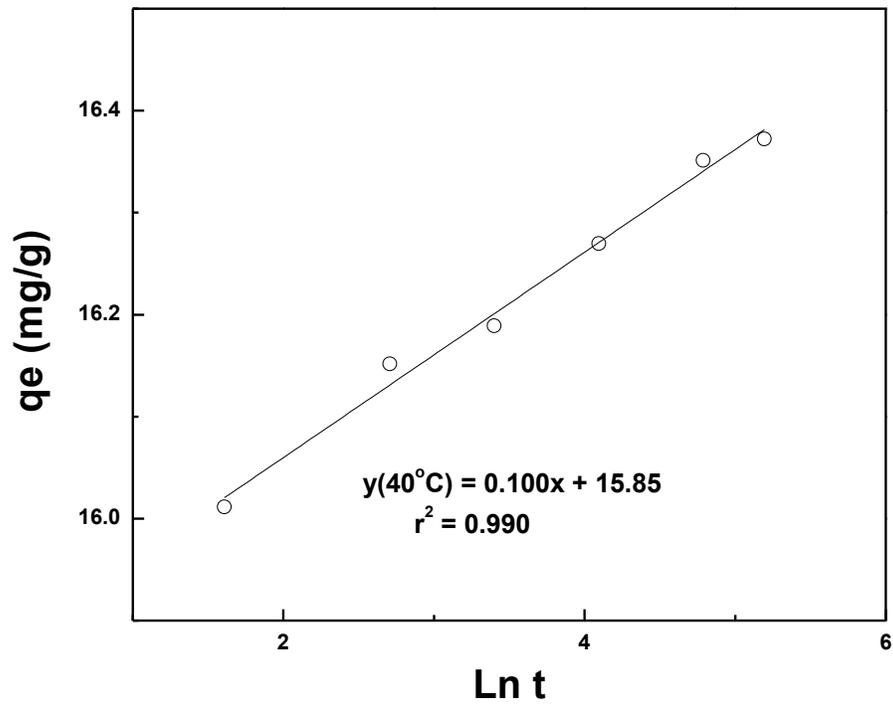


Figure S2c: Elovich kinetics model isotherm at 40 °C (plot of $\log qt$ vs $Ln t$)