

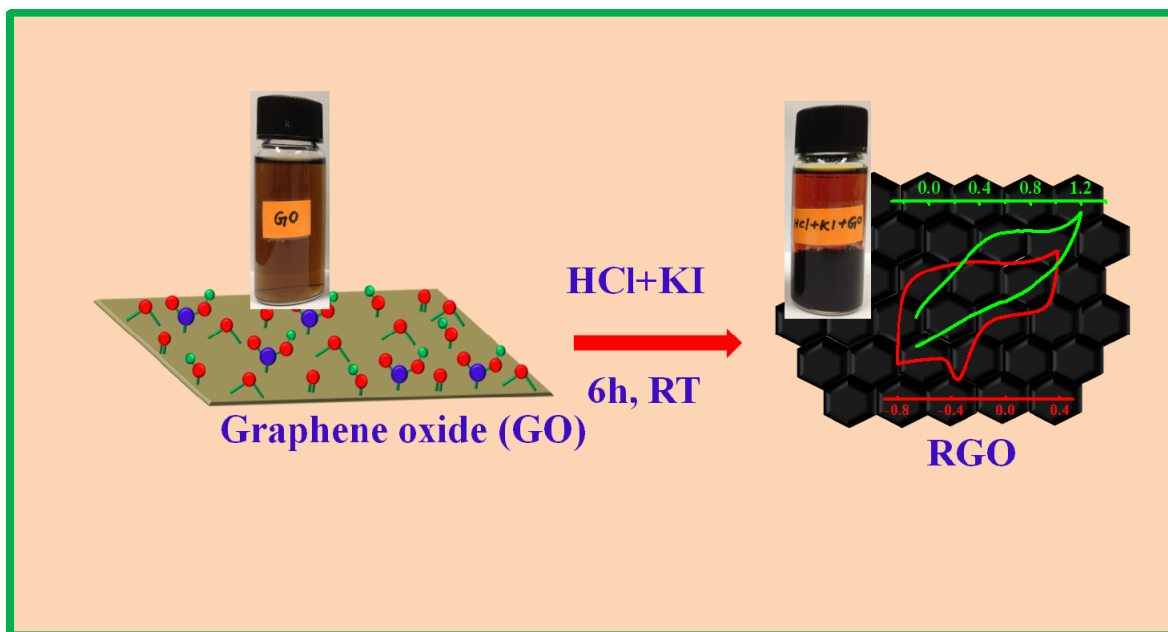
Supporting Information

Iodide-mediated room temperature reduction of graphene oxide: a rapid chemical route for the synthesis of a bifunctional electrocatalyst

Ashok Kumar Das,¹ Manish Srivastav,¹ Rama K. Layek,¹ Md. Elias Uddin,¹ Daeseung Jung,²
Nam Hoon Kim,¹ Joong Hee Lee^{1,2*}

¹Department of BIN Fusion Technology, Chonbuk National University, Jeonju, Jeonbuk, 561-756, Republic of Korea

²Department of Hydrogen and Fuel Cell Engineering, Chonbuk National University, Jeonju, Jeonbuk, 561-756, Republic of Korea



Experimental

1. Preparation of graphene oxide (GO)

Graphene oxide (GO) was synthesized by the modified Hummer's method.¹ In a typical synthesis, 2 g of graphite flakes were placed in a round bottom flask containing 46 mL of concentrated sulfuric acid. To this, 6 g of solid potassium permanganate was added gradually within 30 min at 0–5 °C under vigorous stirring for 2 h. After 2 h of the reaction, the round-bottom flask was transferred to an oil bath pre-heated to 35 °C and stirred overnight. An additional 90 mL of de-ionized (DI) water was added and the reaction mixture was subjected to stirring for another 2 hr. The entire reaction mixture was then poured into a beaker with a 1 L capacity and 35% hydrogen peroxide was added drop-wise until the color of the solution changed to bright yellow. Excess manganese salt present in the GO was removed using dilute hydrochloric acid (5% by volume). After washing several times, the GO was dispersed in water and maintained there until its pH became neutral.

2. Electrode preparation

The glassy carbon (GC) electrode was polished with alumina powder and was subjected to ultra-sonication for 10 min and washed thoroughly with water. The cleaned electrode was then dried in a vacuum oven at room temperature. The RGO (5 mg) was dispersed in a mixture of containing 30 µL of Nafion (3 wt %) in 200 µL of ethanol and ultrasonicated for 30 min. Thirty microlitres of this suspension was pipetted out and drop cast on the GC surface. The electrode was dried in a vacuum at room temperature to obtain a RGO-based electrode.



Fig. S1. Optical images obtained for the GO solution, GO in HCl, in KI, and in a mixture containing HCl and KI after 6 h of the reaction.

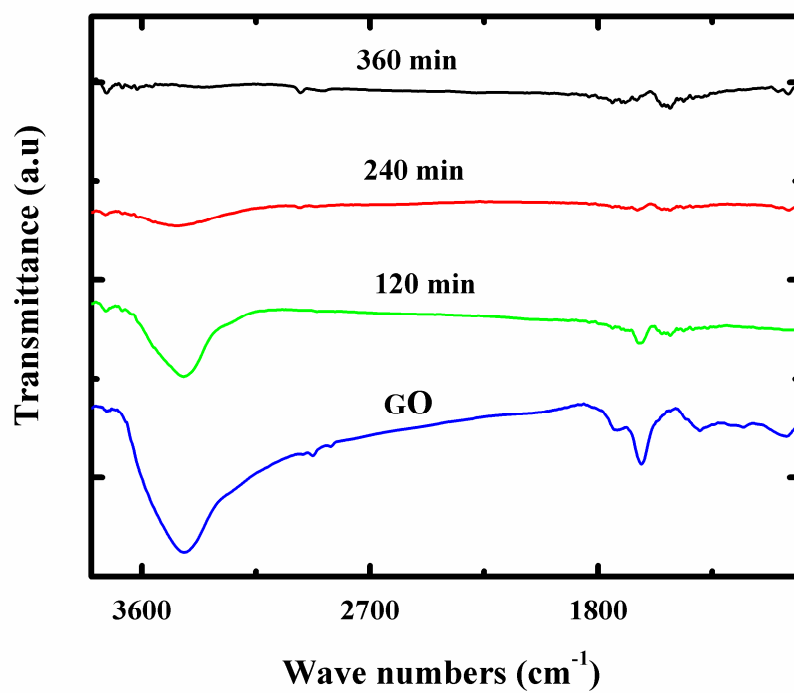


Fig. S2. FTIR spectral profiles recorded at different reduction times of (a) 0, (b) 120, (c) 240 and (d) 360 min.

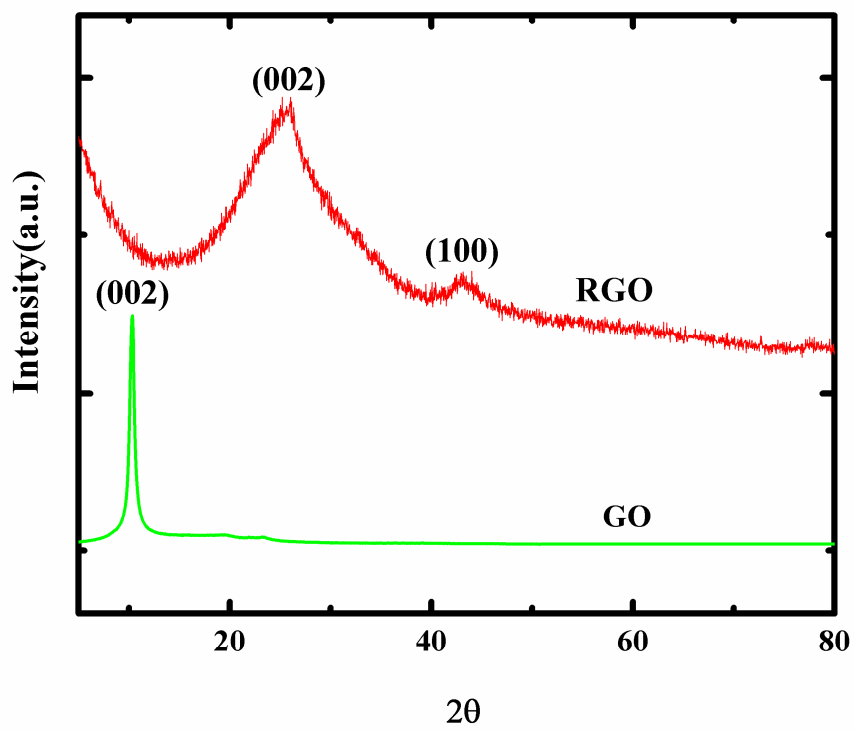


Fig. S3. X-ray powder diffraction patterns obtained for GO and RGO.

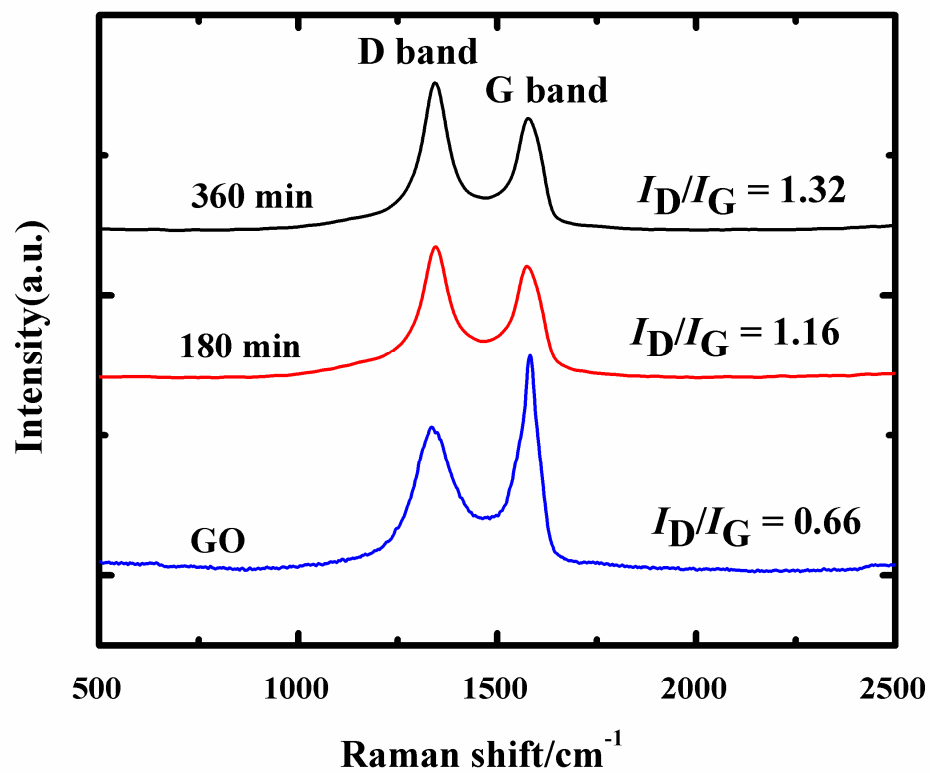


Fig. S4. Time dependent Raman spectra obtained for graphene oxide (GO) reduction using HCl and KI.

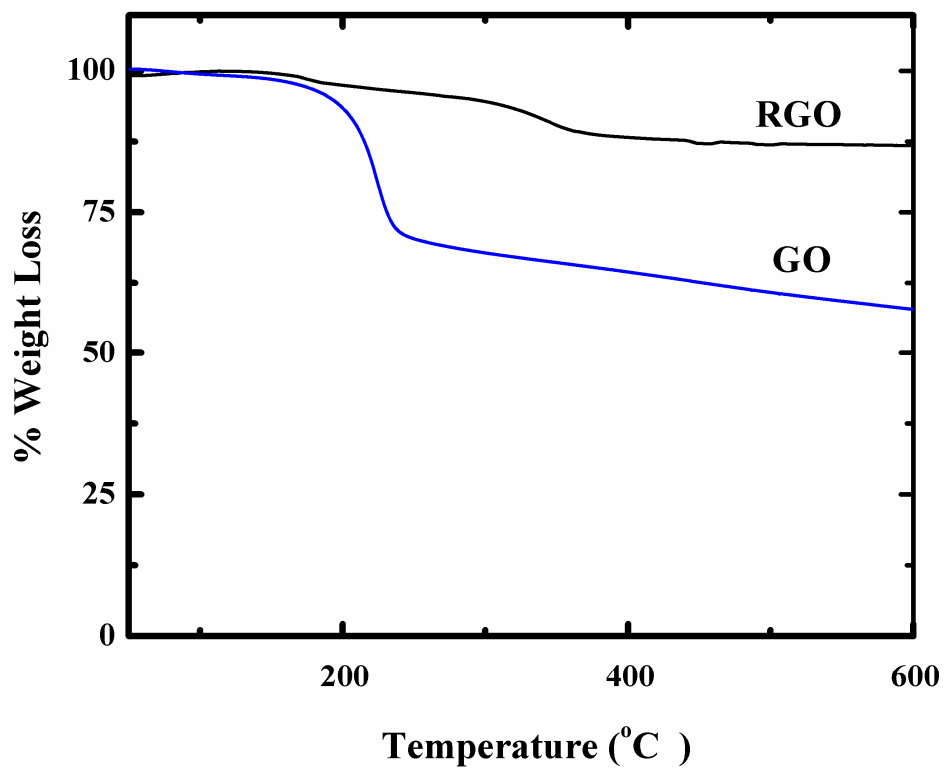


Fig. S5. Normalized TGA plots of graphene oxide (GO) and reduced graphene oxide (RGO).

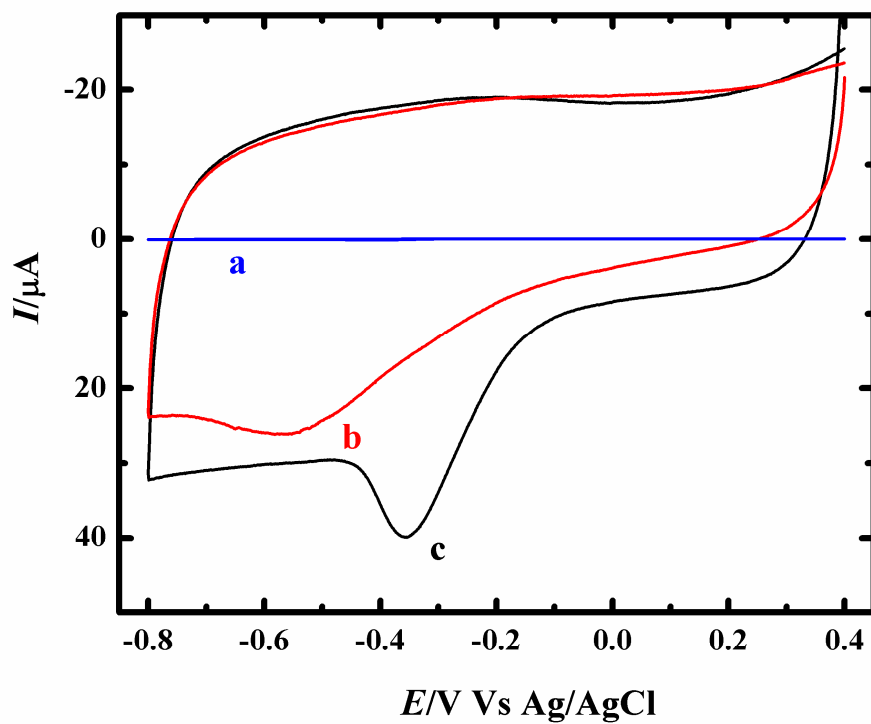


Fig. S6. Cyclic voltammograms obtained for the reduction of oxygen in 0.1M KOH on the (a) GC, (b) NaBH_4 -reduced RGO, and (c) RGO-based electrode synthesized using HCl and KI. Scan rate: 50 mV/s.

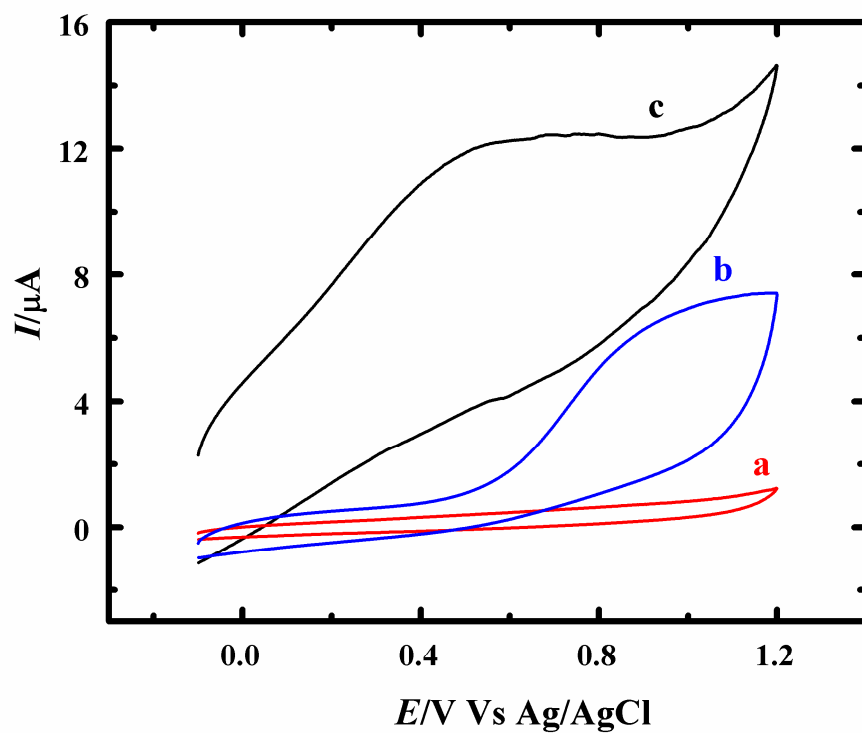


Fig. S7. Cyclic voltammograms obtained for the oxidation of hydrazine in 0.1M KOH on the (a) GC, (b) $NaBH_4$ -reduced RGO, and (c) RGO-based electrode synthesized using HCl and KI. Scan rate: 50 mV/s.

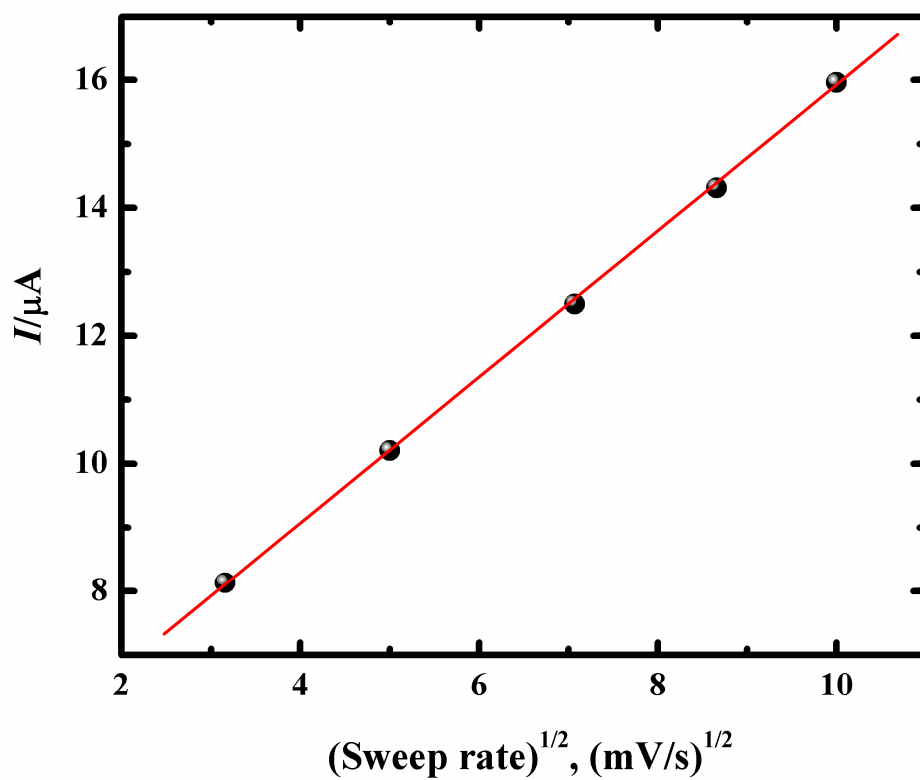


Fig. S8. Plot of the oxidation peak current against the square root of sweep rate obtained for the oxidation of hydrazine on the RGO-based electrode.

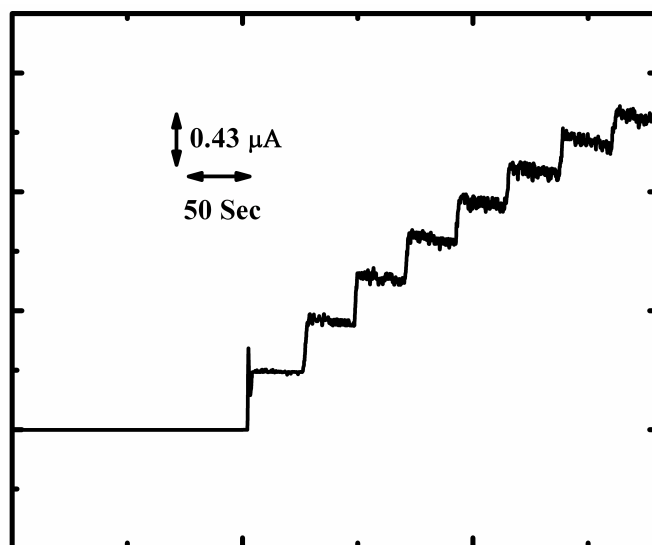


Fig. S9. Amperometric response obtained for the RGO-based electrode towards the sensing of hydrazine in 0.1M KOH. 0.5 mM of hydrazine was injected at regular interval.

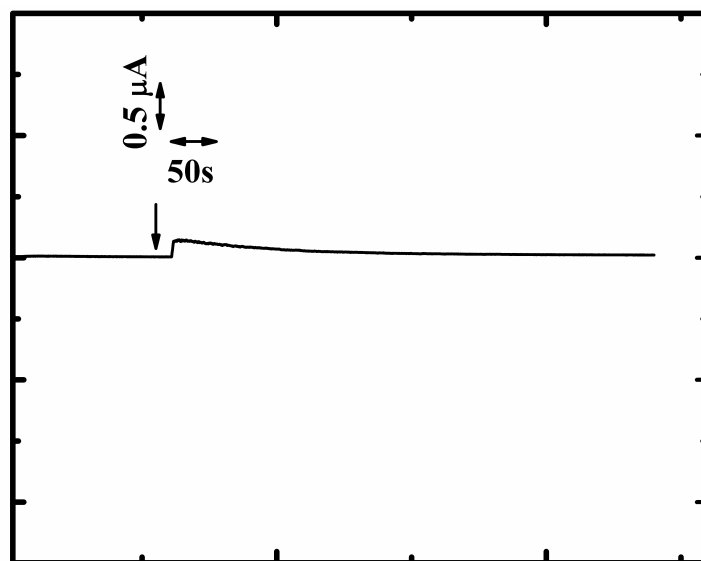


Fig. S10. Operational stability of the RGO-based electrode in 0.1 M KOH containing 1 mM hydrazine.

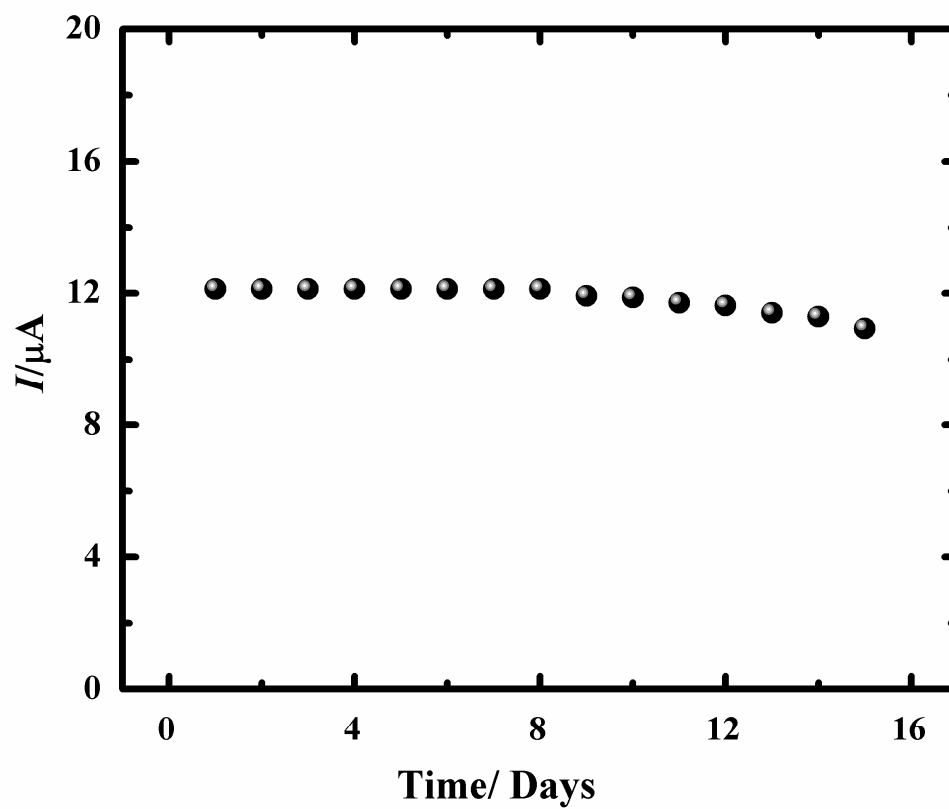


Fig. S11. The long-term storage stability of the RGO-based electrode was examined in 0.1 M KOH containing 15 mM of hydrazine. The electrochemical response of the same electrode was evaluated over the course of 15 days.

References

1. W. Hummers and R. Offeman, *J. Am. Chem. Soc.*, 1958, **80**, 1339