

**Supporting Information for Publication**

**Systematic Study on Morphological, Electrochemical  
Impedance and Electrocatalytic Activity of Graphitic Carbon  
Nitride Modified on Glassy Carbon Substrate from Sequential  
Exfoliation in Water**

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$K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$  containing 0.1 M KCl at scanning frequencies from 0.01 to 100,000 Hz.

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**Table S1.** FT-IR and ATR-FT-IR spectral data of powder GCN and GCN coated on GC plates with different exfoliation time.

**Table S2.** The XPS peaks for GCN powder and GCN coated on GC plate after exfoliation in water for 120 min and their assignments.

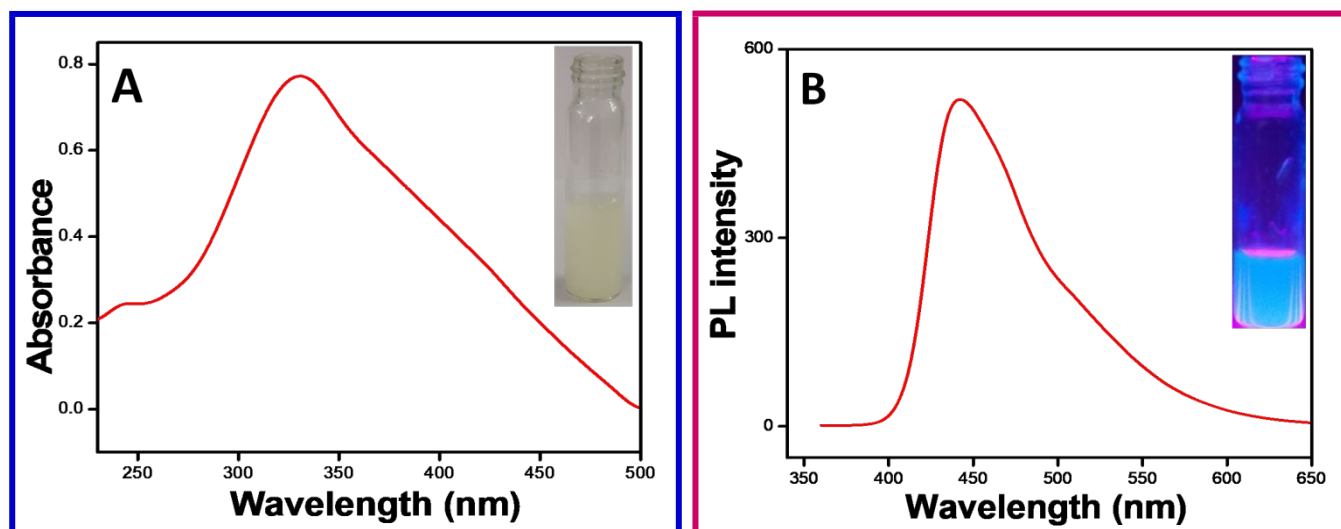
**Table S3.** The electroactive surface area, charge transfer resistance and heterogeneous electron transfer rate constant obtained for bare GC and GCN coated on GC electrode from different exfoliation time.

## Chemicals

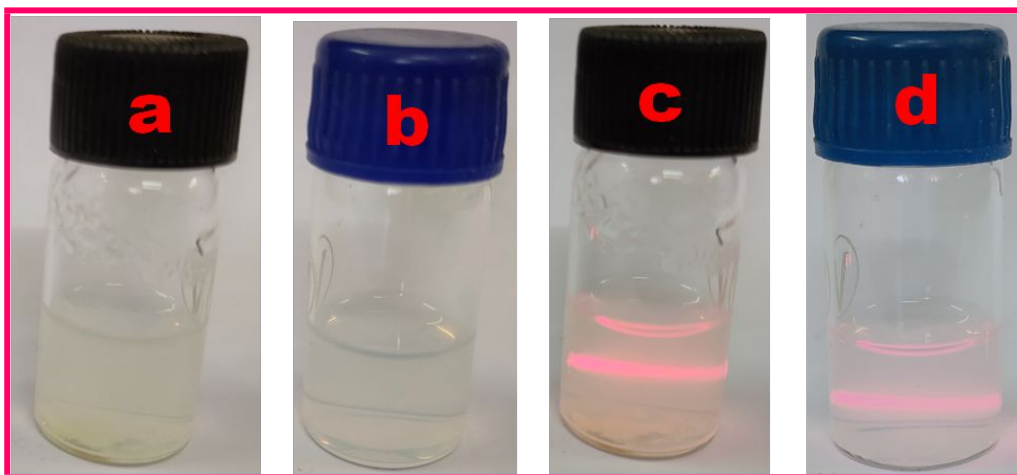
Urea (U), hydrogen peroxide (HP), dopamine (DA), ascorbic acid (AA), sulfuric acid ( $\text{H}_2\text{SO}_4$ ), sodium dihydrogen phosphate dihydrate ( $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ ) and disodium hydrogen phosphate dihydrate ( $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$ ) were purchased from Merck, India and were used as received. Glassy carbon (GC) plates were purchased from Alfa-Aesar.  $\text{Na}_2\text{HPO}_4$  and  $\text{Na}_2\text{H}_2\text{PO}_4$  were used to prepare phosphate buffer (PB) solution (pH 7.2). Double distilled water was used to prepare the solutions used in the present work.

## Instrumentation

Absorption spectra were measured using JASCO V-750 UV-visible spectrophotometer. Emission and excitation spectra were performed on a JASCO-FP-8500 spectrofluorimeter. The FT-IR measurements were carried out in JASCO FT-IR-460 plus model. High resolution transmission electron microscopy (HR-TEM) images were taken from JEOL JEM 2100, USA operating at 200 kV. XPS measurements were done by using PHI 5000 VERSAPROBE scanning ESCA Microscope. Rigaku X-ray diffraction unit using Ni-filtered  $\text{Cu K}\alpha$  ( $\lambda=1.5406\text{\AA}$ ) radiation was used for XRD measurements whereas CHI model 643B (Austin, TX, USA) electrochemical analyzer was used for electrochemical measurements. Scanning electron microscope (SEM) measurements were carried at VEGA3 TESCAN, USA. For ATR-FT-IR, XRD, XPS and SEM analysis GC plate was used as a substrate.

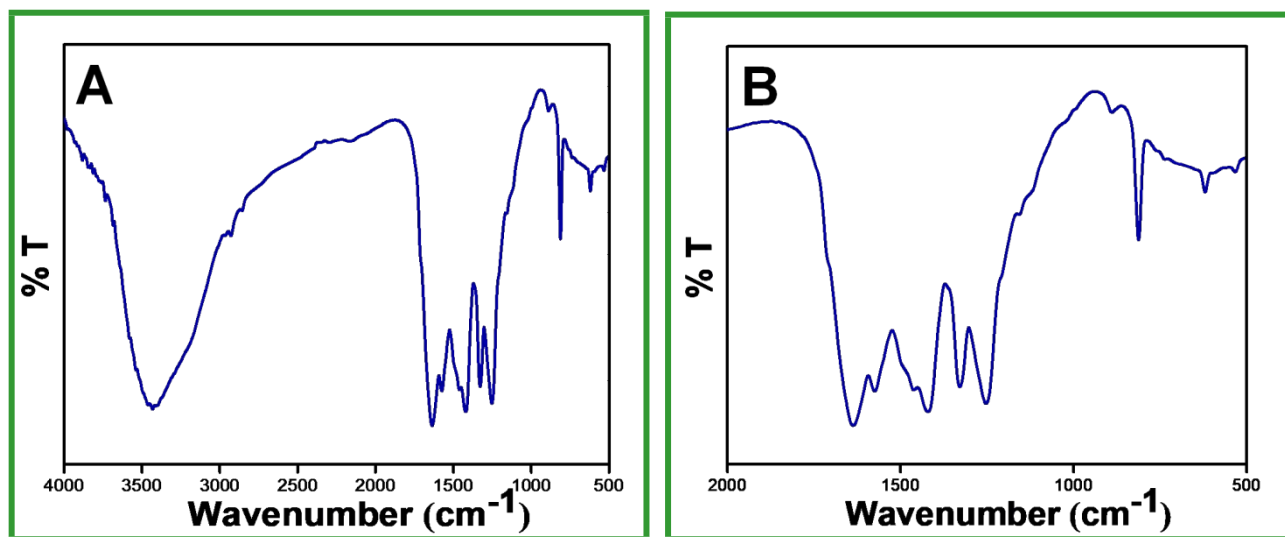


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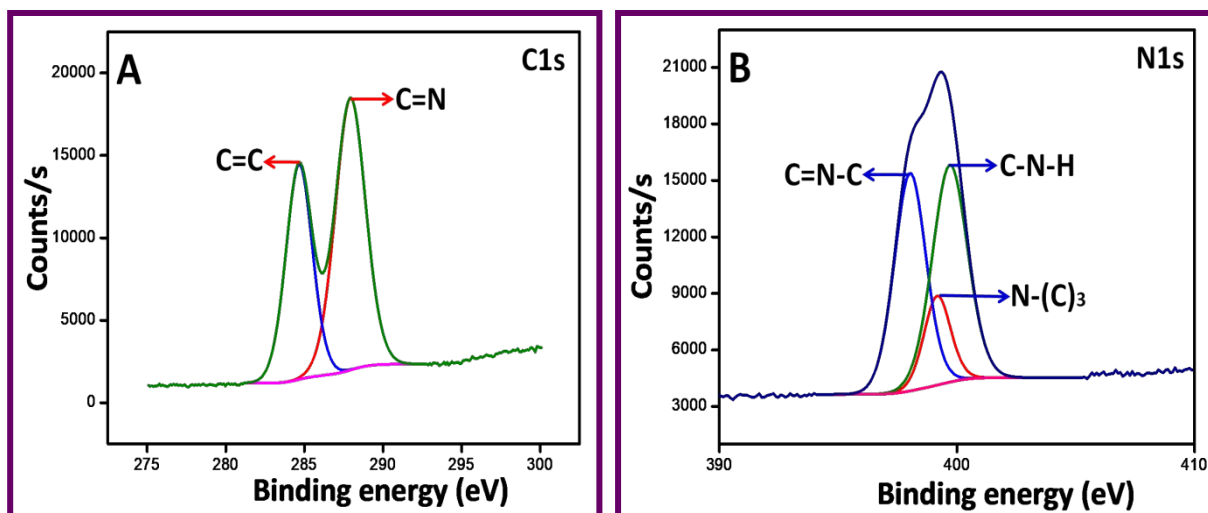


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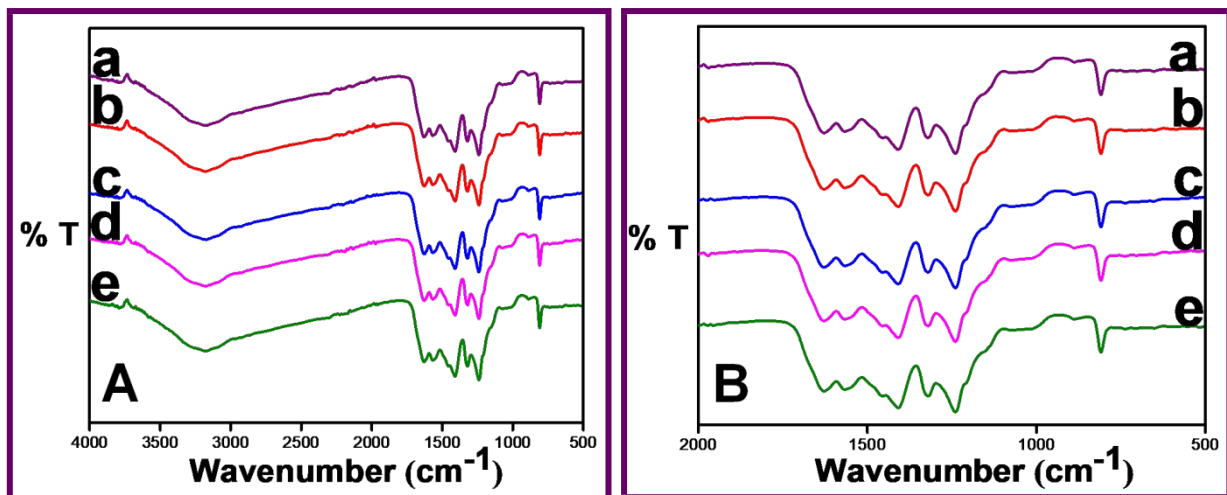


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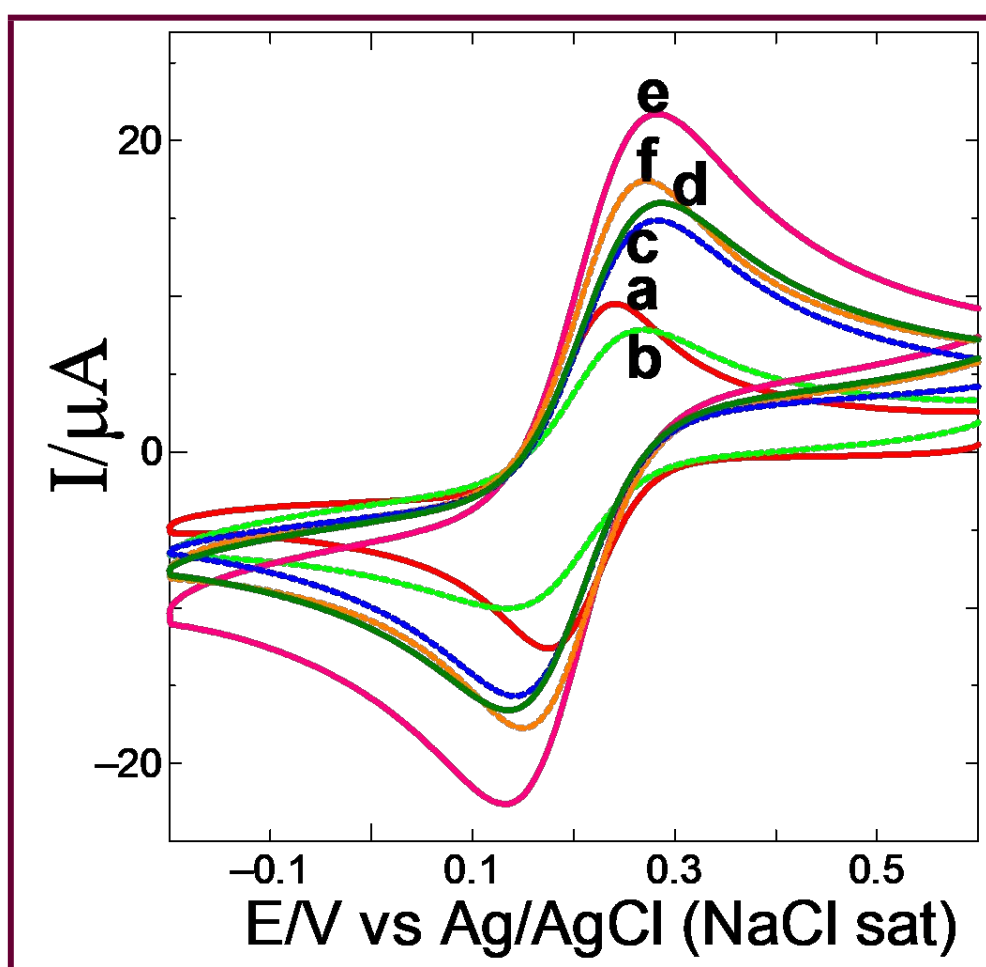


**Figure S4.** XPS of (A) C1s and (B) N1s regions for powder GCN.

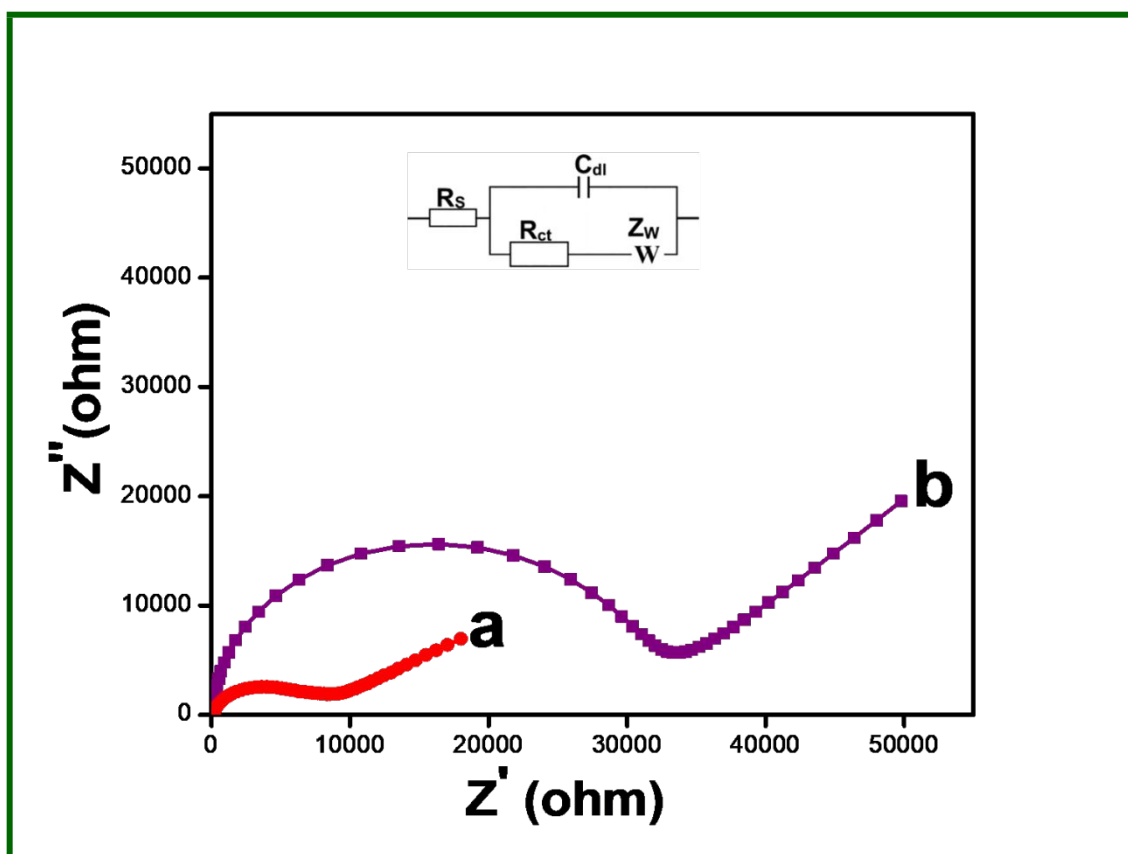




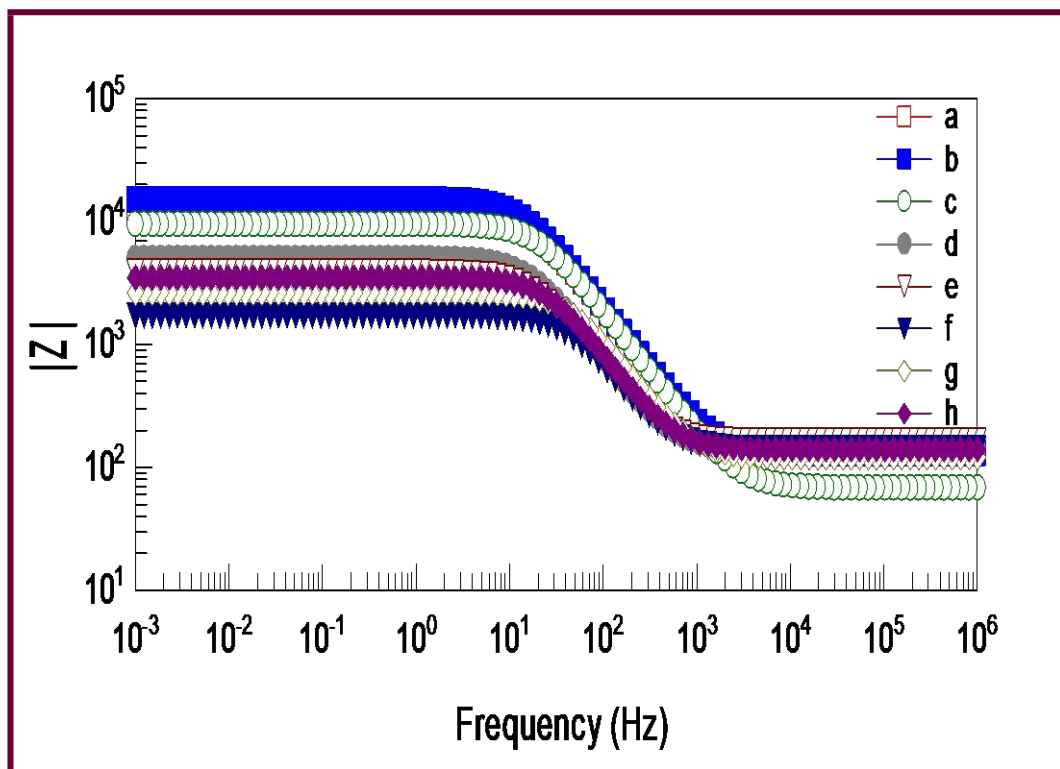
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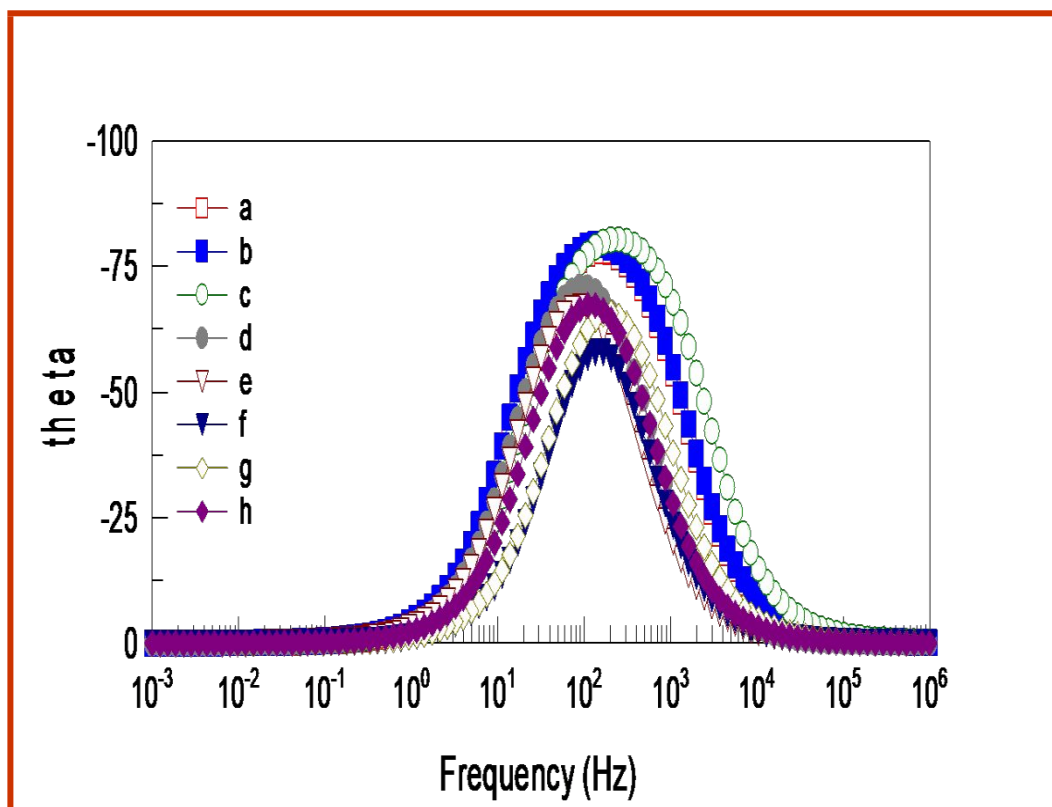
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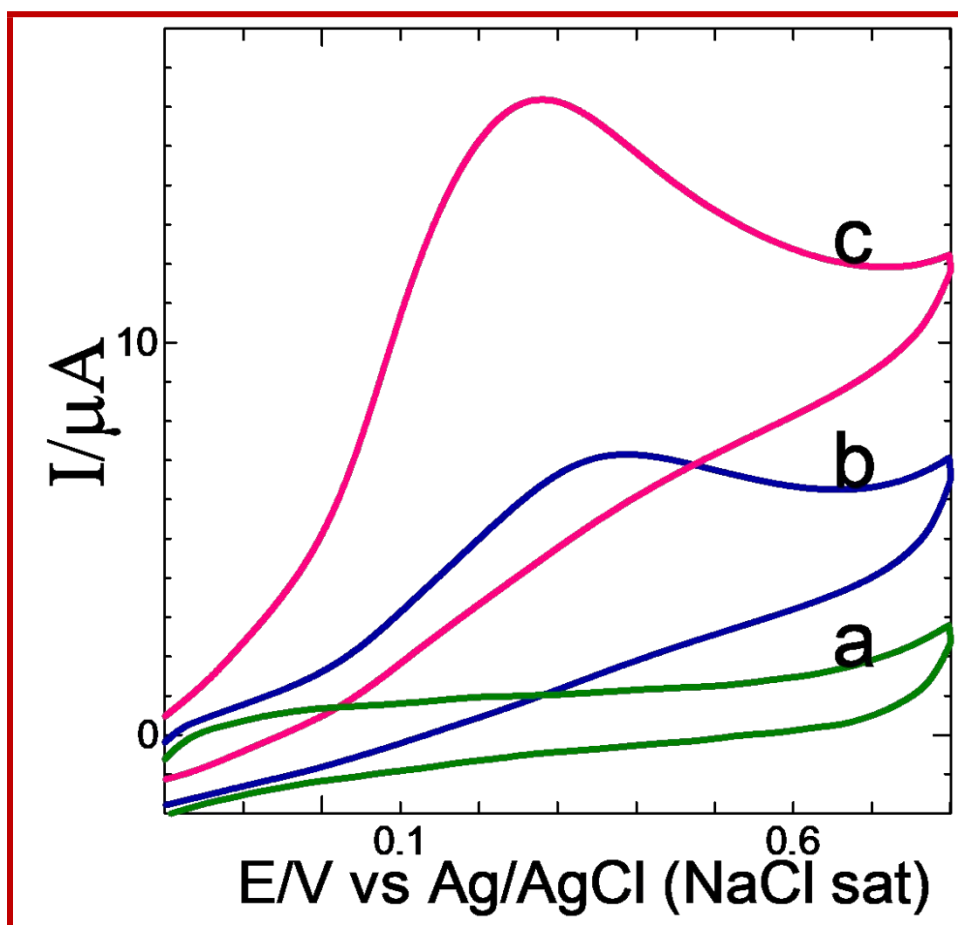
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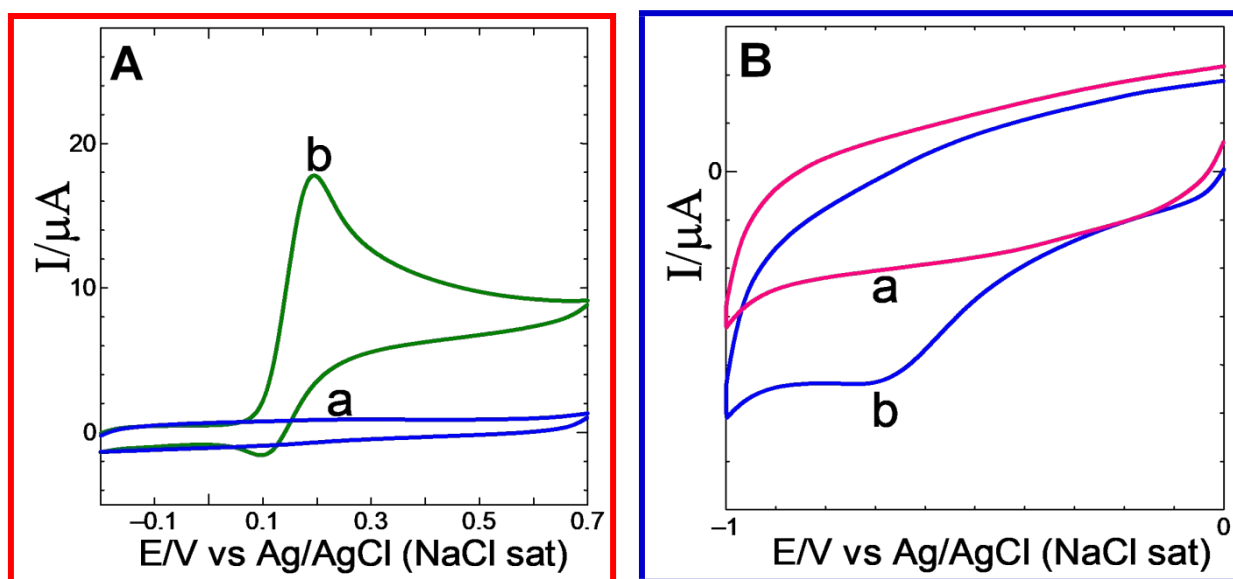
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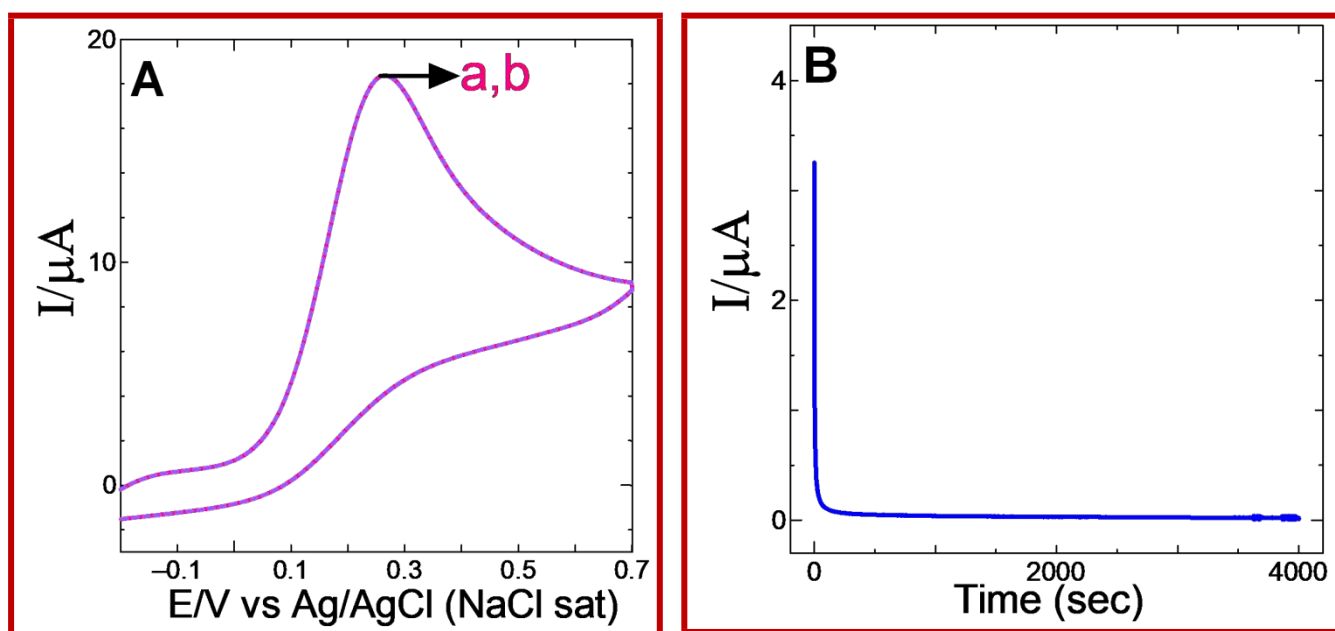
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**Figure S11.** CVs obtained for 1 mM (A) DA and (B) HP at (b) supernatant solution of centrifuged GCN (120 min exfoliation) modified on GC electrode in 0.2 M PB solution (pH 7.2) at a scan rate of  $50 \text{ mV s}^{-1}$ . (a) Supernatant solution of centrifuged GCN (120 min exfoliation) modified on GC electrode in the absence of (A) DA and (B) HP.



**Figure S12.** (A) CVs recorded for the oxidation of 1mM of AA at GC electrode modified with GCN (120 min exfoliation) in 0.2 M PB solution pH (7.2) at different cycles after (a) 1<sup>st</sup> and (b) 10<sup>th</sup> cycles. (B) Chronoamperometric *i-t* curve for GC electrode modified with GCN (120 min exfoliation) in 0.2 M PB solution (pH 7.2).



Band positions (cm <sup>-1</sup> )						Peak assignments
GCN powder	GC/GCN (15 min)	GC/GCN (30 min)	GC/GCN (60 min)	GC/GCN (120 min)	GC/GCN (180 min)	
3431	3172	3180	3172	3181	3182	N-H stretching
1636, 1574	1628, 1566	1628, 1566	1628, 1566	1628, 1566	1628, 1566	C=N
1461, 1421, 1329, 1252	1455, 1320, 1240	1455, 1320, 1240	1455, 1320, 1240	1455, 1320, 1240	1455, 1320, 1240	C-N
810	808	808	808	808	808	Breathing mode of the triazine unit

**Table S1.** FT-IR and ATR-FT-IR spectral data of powder GCN and GCN coated on GC plates with different exfoliation time.

Binding energy (eV)		Peak assignments
GCN	GC/GCN	
284.7 288.4	284.6 288.3	<b>C 1s</b> C=C sp <sup>3</sup> C=N
398.5 399.3 400.3	398.7 399.5 400.7	<b>N 1s</b> sp <sup>2</sup> C=N-C N-(C) <sub>3</sub> C-N-H

**Table S2.** The XPS peaks for GCN powder and GCN coated on GC plate after exfoliation in water for 120 min and their assignments.

Parameters	Exfoliation time (min)	EASA (cm <sup>2</sup> )	R <sub>CT</sub> (Ω)	<i>k</i> <sub>et</sub> (10 <sup>-4</sup> )
Bare GC	-	0.06	14916	2.3
GC/GCN	15	0.04	22370	1.7
GC/GCN	30	0.05	19524	1.8
GC/GCN	60	0.09	7092	5.5
GC/GCN	90	0.23	5010	7.6
GC/GCN	120	0.35	1816	20.9
GC/GCN	150	0.28	3248	11.7
GC/GCN	180	0.26	4988	7.6

**Table S3.** The electroactive surface area, charge transfer resistance and heterogeneous electron transfer rate constant obtained for bare GC and GCN coated on GC electrode from different exfoliation time.