#### Supporting information

# Synthesis and characterization of conductive few layer graphene nanosheets using an anionic electrochemical intercalation and exfoliation technique

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### 1. Drawback of Electrochemical exfoliation at constant potential:

### 1.1. Experiment:

Prior to constant applied potential electrochemical exfoliation, all the pre-treatment experimental procedure was followed as per our standard method discussed in the main context. Then the cathodically pre-treated graphite electrode was connected as anode in the electrolytic bath at a constant potential 8 V. After that the exfoliated graphite flakes were collected from the electrolytic bath and rinsed off in double distilled water to remove any un-dissociated and ionic form of HClO<sub>4</sub> content. The dispersed product was then subjected to probe ultra-sonication for four hours to further disintegrate the graphitic flakes. The homogeneously dispersed sonicated graphene colloidal is centrifuged at 6000 rpm for 45 minutes and the decant is considered as FLGNSs. The constant applied potential electrochemical exfoliation process were carried out in four different electrolytic concentration namely 0.5, 1.0, 1.5 and 2.0 M HClO<sub>4</sub> and the respective exfoliated product named as P1, P2, P3 and P4.

#### 1.2. Visual Inspection:

Figure S1 describe the comparative visual analysis of the exfoliation process and nature of the products obtained between direct and ramping bias. Fig. S1(a) shows the rapid exfoliation of the graphite sheets with heavy graphite flake which were immediate settled down at the bottom of the reactor where as there is a fine dispersion of light weighed graphene flake forming a black ribbon type band around upper part of the electrolyte with clear distinct of heavy carbon flake settled at the bottom obtained by ramping bias as shown in Fig. S1(b). Very similar results were obtained in all four different electrolytic concentration of exfoliation at constant applied bias at 8 V, which can be seen form Fig. S1(c). After three week of study, the exfoliated product obtained by ramping bias upto 8 V has not remarkable settled can be depicted from Fig. S1(d). Thus, the electrochemical exfoliation done at ramping bias may give enough time for wetting and intercalation to the graphite sheet. As a result of this smooth exfoliation occurs at the anodic side for obtaining light weighed graphite flake which can be further sonicated and centrifuged for achieving FLGNSs.



Fig. S1. Electrochemical exfoliation of pyrolytic graphite sheet in aq.  $HCIO_4$  at constant 8 V (a), at ramping rate upto 8 V (b), exfoliated product at constant applied potential of 8 V in four different electrolytic conditions (i.e., 0.5, 1.0, 1.5 and 2.0 M aq.  $HCIO_4$ ; labeled as P1, P2, P3 and P3 respectively) (c), and comparative visual inspection of dispersing ability between constant and ramping applied potential exfoliated product after 3 week (d).

#### 1.1.3. Morphological analysis:

Furthermore, morphological analyses were carried out for the as-synthesised electrochemically exfoliated product obtained by constant applied bias at 8 V as shown in Fig. S2. From the FESEM and TEM image analyses, it is found the electrochemical exfoliated graphite flakes are not fully exfoliated layer by layer. Shock bias at high potential degrades the quality of the exfoliated product.







Fig-S2. SEM and TEM images of electrochemically exfoliated graphite flake obtained at P1, P2, P3 and P4 electrolytic condition upon constant applied bias of 8 V.

## 1.4 X-ray photoelectron spectroscopy (XPS) analysis:



Fig-S3. XPS analysis of various functional groups bonded to the FLGNSs obtained by electrochemically exfoliation at the electrolytic bath conditions of PA3 (a) and PA4 (b).