# Facile Single Step Electrochemical Synthesis of Few Layered Graphene for energy storage Applications

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**Abstract**

Recently developmentof the large-scale synthesis was few layered graphene (FLG) via electrochemical exfoliation (EC) method. The few graphene sheets (EC-FLG) were characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM), High resolution Transmission electron microscopy (HR-TEM). The electrochemical analysis showed that an enhanced storage and capacitance performance of the few layer graphene was obtained. The electrochemical measurements of cyclic voltammetry (CV) were for Electric double-layer capacitor (EDLC)energy storage for supercapacitors as observed specific capacitance calculations (Csp) –107 F/g scan rate at 100 mV/s. The electrochemical exfoliation techniques to find great promise for the industrial-scale synthesis of high-quality defect free graphene for energy storage advanced applications.

**Keywords**: Graphene, Electrochemical method, exfoliation, energy storage, Supercapacitors.

## 1. INTRODUCTION

In recent years graphene is consistedto the mono layer carbon atoms arranged densely and closely honeycomb like hexagonal structure with 2D nanostructure materials [1]. Graphene was focused various applications in future electronics, electrochemical energy storage and energy conversation devices, due to their good electrical properties, high thermal stability, excellent mechanical strength and high surface area. Commonly followed synthesis methods of few as well as single layer of graphene chemically exfoliation methods modified hummer's method, chemical vapor deposition and microwave method [2, 3]. Among these methods are some cost wise expensive and some limitations but recently focused electrochemical methods is simple and coast effective techniques electrochemical exfoliations from raw bulk graphite sources [4-9]. Carbon based materials such as CNTs and Graphene are the most promising application in supercapacitor electrode materials for high energy storage devices in first explored by Ruoff and coworkers. The supercapacitor also called electrical double layer capacitor high energy storage. As a remarkable focused Supercapacitors light weight, miniaturized size, high stability, high specific capacitance and high-power density and energy density compared to Li-Ion batteries [10 & 11]. In this paper we are developed novel method of graphene, we are developed and introduced electrochemical exfoliation in aqueous ionic liquid. Few layer graphene nanosheets were single step large scale production from high pure bulk graphite rods. The EC-FLG material was characterized by XRD, SEM, TEM and Electrochemical analysis.

## 2. EXPERIMENTAL

### 2.1 Single Step Synthesis of few Layer Graphene

The few layer graphene sheets were synthesized by electrochemical exfoliation from graphite source. In the experimental detailed: Both as anode and cathode materials were using high pure graphite rods (Sigma Aldrich, 99% Grade length 8 cm, dia 0.5 cm) were inserted into the ionic aqueous electrolytes. The synthesis of electrochemical intercalation role of aqueous electrolyte ($H_2SO_4$) graphite into reduced few layer exfoliation graphene nanosheets. The aqueous ionic electrolyte was 10ml Sulfuric acid ($H_2SO_4$) and 20ml D.I waterinto200 mlbeaker.Above mixed solution stirring vigorously at30min as electrolyte solution. The constant direct current (DC) Power source meter was used. High pure graphite electrodes were connected in electric power clip and both rods were dipped into the electrolyte solution. The reaction was carried out constant DC was applied 5Vin betweenthe pure graphite electrodes. The reaction was carried out 2 hrs. When the reaction start the anodic graphite rod was slowly exfoliated and floated and suspended fine dark black colored solution appearance high quality graphene sheets. When the electrode is starting exfoliation very slowly anodic erosion graphene Nanosheets particles suspended into the electrolyte solution. Finally, the purifications process is centrifugedand used D.I water to removed the acetic nature into neutral (pH=7). The Fine block power dried overnight and annealed muffle furnace 400 °C for 5h. The final product graphene nano sheets goes to further materials analysis.

### 2.2 Electrochemical Analysis

The EC-few layer Graphene were analyzed Electrochemical Instrumentation (Metrohm-Autolab, PGSTATE 302N) in a three electrodes cell system, consists a S.S Mesh electrode (stainless steel-mesh) as working electrode, Pt wire as counter electrode and Ag/AgCl as a reference electrode with 1M $H_2SO_4$ electrolyte solution. Cyclic voltammetry study was carried out in CHI760D model in the potential range of -0.2 –0.6 V with various scan rates of 10, 20, 30, 40, 50 and 100 mV/s.

## 3. RESULT AND DISCUSSIONS

### 3.1 XRD, SEM & TEM

The EC-FLG was characterized by XRD pattern shown in Fig. 1 (a) after graphite electrode was exfoliated shows up a peak at around 26.6° corresponding plane to the (002) which indicates an interlayer spacing of 3.34 Å. After electrochemical exfoliation diffracted peak at 26.6° in becomes broad peak can be attributed to the exfoliation of layered structures of graphite. The broad peak reveals that the partial restacking of exfoliated graphene layers. SEM analysis was carried out and is shown in Fig. 1(b) & (c). The electrochemical exfoliated as prepared few layer graphene nanosheets for supercapacitor electrode, to confirm the few layers one by one staked with bended graphene sheets. From the SEM micro graph images it is clear that all the morphological sheets, its thickness around 60-80 nm. In order to evaluate their exfoliation layers few sheets were obtained due to the constant potential with ionic intercalate will be applied between electrodes.

HR-TEM analysis for EC-FLG (nanometers scale) was surface morphological was observed to be placed graphene nanosheets edges fine each other. The few graphene nano layer transparency regions indicated by arrows in block aero marks Fig.1 (d) wasreveal thatthe few layer graphene nanosheets.

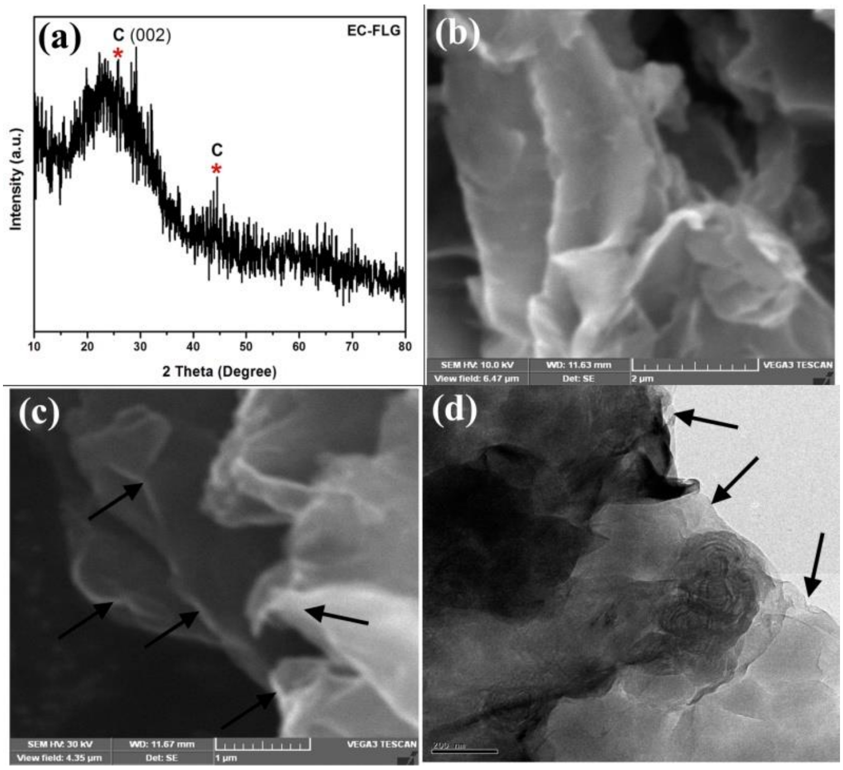


**Fig. 1(a) XRD pattern of EC-FLG, (b & c) SEM images of EC-FLG (d) HR-TEM images of EC-FLG**

A possible mechanism for the intercalation process has been proposed for electrochemically exfoliation process for the mechanism of electrostatic forces interacts with ions graphite layers exfoliate, the electrolyte containing with electrochemically Intercalation process of $H_2SO_4$ acid $H^+$ cation and $SO_4^{2-}$ anion with bulk graphite exfoliate layer by layer exfoliation to observe few layer graphene sheets like transparent morphology.

### 3.2 Cyclic Voltammetry

The cyclic voltammetry (CV) of EC-FLG at various scan rates of 10, 20, 30, 40, 50 and 100 mV/s in the potential range of -0.2–0.6 V are shown in Fig. 2 (a). As can be observed from the CVs of EC-FLG, with the increase in scan rate the current also increases which. The specific capacitance ($C_{sp}$), was calculated from cyclic voltammetry (CV) curve was using at scan rate 100 mV/s obtained Specific capacitance $C_{sp}$ is 107 F/g for EC-FLG using the formula $C_{sp}$ = integrated total area of the CV curve/ (Scan rate * total Voltage window * mass of the active material). From Fig. 2(b). The CV cure $C_{sp}$ was calculation using 100 mV/s for rectangular shape for Electric/ electrochemical double layer capacitive (EDLC) nature.

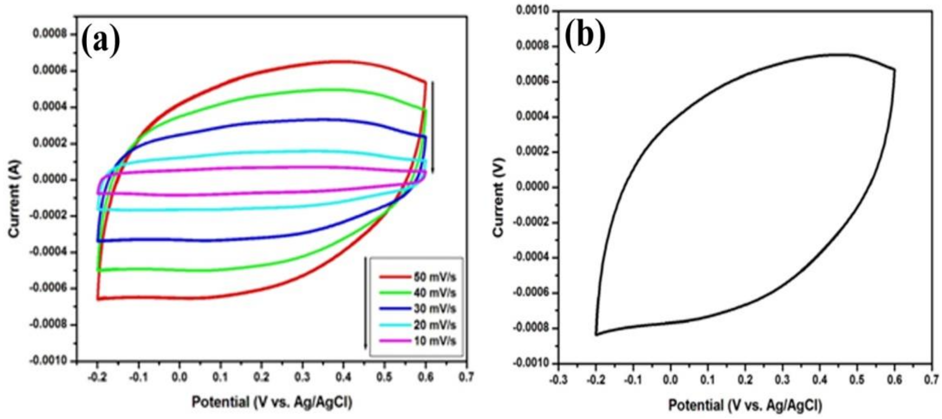


**Fig. 2:**
**(a) Cyclic voltammetry of T-RGO at different sacn rates (10 - 50 mV/s)**
**(b) Cyclic voltammetry of T-RGO at scan rate 100 mV/s.**

## 4. CONCLUSION

A facile synthesis of few layered graphene sheets was developed in bulk graphite electrode. This method was found that produced high yield with good quality graphene. The EC-FLG material was characterized for structural and morphological analysis of powder XRD, SEM and HR-TEM observation. The electrochemical exfoliated graphene for used assupercapacitor electrode. From the CV curve usedto found that maximum specific capacitance was calculated (Csp) –107 F/g at 100 mV/s for EC-FLG material. Electrochemical few layer defect free graphene nanosheets was possible to produce by large scale. This work observation confirmed the few layer defect free graphene nanosheets and also demonstrated possible in future energy storage materials for supercapacitor device fabrications.

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