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Synthesis of Few Layer Graphene Using Mechanical Exfoliation of Kitchen Blender Method for Super Capacitor Applications

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Abstract

Facile and single step preparation of few layer graphene (FLG) by mechanical exploitation kitchen blender method. The obtained few layered graphene was characterized by FTIR, XRD, SEM, HR-TEM. The morphology images of SEM and HR-TEM was high-quality and defects free. The electrochemical measurements of cyclic voltammetry (CV) revealed that Electric double-layer capacitor (EDLC). The charge discharge measurements from specific capacitance was calculated (Csp) – 33 F/g at current density 1 A/g. The Electrochemical impedance spectroscopy (EIS) performed was charge transfer resistance (Rct) value for 8.57 ohm. Few layer graphene was obtained from simple kitchen method. This method active material for supercapacitors applications were observed in first time reported. The kitchen blender mechanical exfoliation techniques shows to great promising future industrial-scale synthesis of high-quality defect free graphene for energy storage device applications.

Keywords: Graphene, kitchen blender method, Mechanical exfoliation, energy storage, Supercapacitors.

1. Introduction

The recent development of two-dimensional (2D) nature materials in interest of research society on this material is continuously growing materialistic world and recent innovation by micro mechanical exfoliation led to the synthesis of single layer graphene [1,2]. The advancing 2D carbon nano structured of graphene research field is majorly focused by the interest of society towards unique and amazing properties possesses by graphene. Due to the strong mechanical, electronics, electrochemical, physical properties of graphene, it has high potential for application as energy storage and conversion applications, electronic devices, and biological applications [2-6]. In the next decade, graphene will find commercial applications in many areas but researchers focus electrical energy storage applications as important, such as graphene based supercapacitor applications. Hence, large scale energy storage electrode active materials will require industrial scale production of defect free graphene in desired form bulk graphite to few layered graphene sheets. Widely, used as a synthesis of graphene as different methods Chemical oxidation [7], physical methods [8]. Among, these methods were producing shown to produce some defects, long time process, purification, low yield graphene. However, no scalable method exists to give large quantities of graphene that is also defect-free. At present, the mechanical exploitation or cleavage method has emerged to be a scalable route to cost wise graphene-based few sheets in considerable quantities for industrializes production [9-11]. The large scale industrial production by kitchen mechanical exfoliation for Energy storage electrodes will almost certainly be require large quantities of graphene in the form of liquid suspensions. While, large scale exfoliated graphene were proved very useful in applications for energy storage device applications.

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In this paper, preparation of defects free graphene single step methods by kitchen mixer mechanical exfoliating tool can be used as for bulk graphite. The few layer graphene for economically commercial industrial scale production. In this method large-scale production graphene used as supercapacitor application, first time reported. The FLG materials were characterized and electrochemical analysis supercapacitor applications in three electrode system.

2. Experimental

The few layered graphene (FLG) was synthesized by mechanical kitchen blender method. In detailed mechanically kitchen blender as new process of exfoliation of graphene by mixing in 5 litter large vessel 5 blade blender above mixed a co-mixing of 5 gram graphite powder (purchased from three different suppliers, Aldrich) (7 – 20 μm) and home used soap liquid few drops in a mixing volume of 800 ml with ultrapure water (Millipore). The5 liters mixture head vessel with speed increased gradually until the desired speed was reached 5000 rpm. The process will start switched on the mixer blender initial speed blade rotating 1000 rpm. The main controls of mixing/exfoliated parameters speed 1000 rpm to maximum speed 5000rpm and whole exfoliation process time for 40 mints. The vessel inside solution volume has increased so original volume after every paused interval (3 mints). The resulting FLG liquid samples are through multiple filtrations with followed by centrifugation steps to obtain FLG dispersions with high pure water (Millipore). The resulting exfoliated large scale few layer graphene sheets powder of dried at 100 °C for 12 h. The collected fine black colored powder few layer graphene by the kitchen blender will be further materials characterizations and electrochemical applications.

2.1. Characterization

The materials were characterized graphite and Few Layer Graphene (FLG), crystal structures and planes were determined by high angle X–ray diffraction (XRD) system using X'PERT-PRO model. Laser Raman spectroscopy was performed using Princeton instruments Acton (SP–2500) with 514.5 nm laser. The surface morphology of graphite and FLG of the samples were characterized by using scanning electron microscope (SEM) with VEGA–3 TESCAN instrument and high resolution transmission electron microscope (HR–TEM) with TECNAI, T–30, operated at 250 KV. The Fourier transformation infrared spectroscopy (FT–IR) spectra were performed by using Model JEOL–234.

3. Result and Discussion

3.1. X-Ray Diffraction (XRD)

The X–ray diffraction pattern of purified (MWCNTs) is shown in the Figure 2. The pattern shows the broad diffracted peaks appears at $2\theta=25.9^{\circ}$ was assigned correspond to (002) plane. The diffraction peak corresponds to (002) plane and d-spacing value 3.39 Å. From XRD pattern shows for MWCNTs the results reveals that the carbon nanotubes with hexagonal structure with carbon phase and diffraction patterns typically well graphitized.

2.2. Electrochemical Measurements

The working electrode was prepared by adding the FLG active materials, the total weight ratio of the Material (mass ratio 80:15:5) well mixed and the solid paste like mixture was casted by placing stainless steel (S.S) mesh as working electrode. Working, Reference & Counter electrodes were used corresponding to the (S.S) mesh, Ag/ AgCl & Pt wire, respectively with1 M H_2SO_4 aqueous solution was

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used as the electrolyte. The capacitive performance was tested on a High Performance electrochemical workstation Autolab (PGSTAT-302N). Cyclic voltammetry (CV) and impedance and galvanostatic charge-discharge was carried out a by three-electrode system electrochemical setup.

3. Results and discussion

3.1. XRD and RAMAN

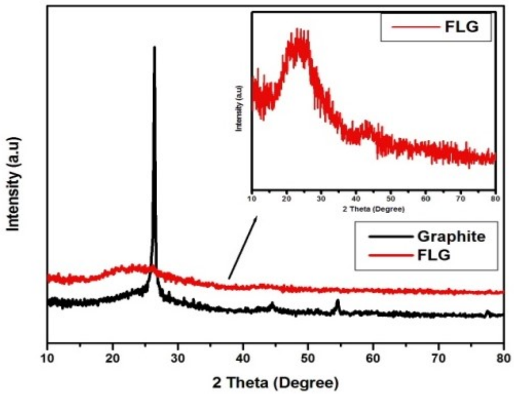

Figure 1. the XRD patterns of FLG

The X-ray diffraction (XRD) pattern Figure 1 shows a single-phased graphite and Few Layer Graphene (FLG). From the X–ray diffraction pattern with different plane values graphite and few layer graphene diffracted peaks at 26º (d-spacing value 3.42 Å) and 26.36º (d-spacing value 3.37Å) are assigned to the (002) planes, respectively. Compare to the graphite peak value, the FLG broad diffraction peak at 25.6º could be related to the (002) plane of the hexagonal structure.

3.2. XRD and RAMAN

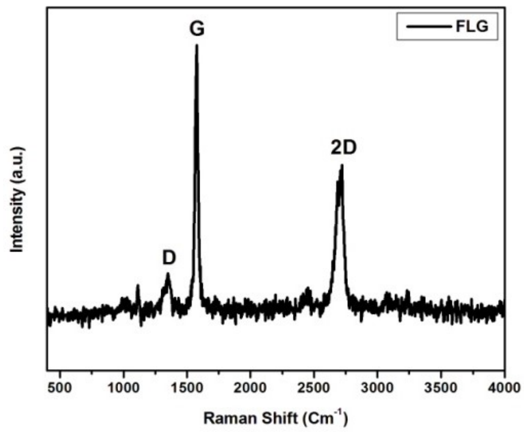

Figure 2. Raman spectra of FLG

As shown the Raman spectra in Figure 2, the Graphite and FLG were significant order and disorders can be characterized the intensity of the D band (1346 cm^{-1}) relative to the G band (1578 cm^{-1}). Compare to G band and D band slightly due to small disorder. Intensity of order/ disorder I_D/I_G = 0.17, its low disorder ratio less than 1, From Raman spectra 2D (2723 cm^{-1}) band indicating that clearly visible few layer graphene indicated and basal plane defects free are introduced during the kitchen blender-assisted exfoliation.

3.2. SEM and TEM

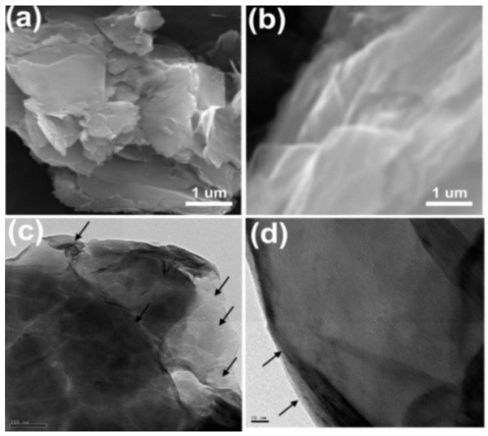

Figure 3.(a) Graphite and (b) SEM images of FLG. (c and d) HR-TEM of FLG

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The surface morphological images are analyzed by SEM and HR-TEM analysis and the images are presented in Figure 3. Figure 3(a) shows the SEM image of graphite bulk particles. Fig 3(b) clearly shows after exfoliations very fine few layer sheets. The HR-TEM images of Figure 3(c) and 3(d) shows demonstrated the free stacked few layer Graphene sheet and the thicknesses of the Graphene nano sheets are around 20 nm. These results revels that Few layers indicates (aero mark) that a particular in edges of the sheets with some layers are overlapped for the mechanical shear exfoliation of Graphene in graphite. Fig 3 (d) defect free surface area morphology and edges of the sheets few layer kitchen blender method from graphite source.

3.3. FT-IR

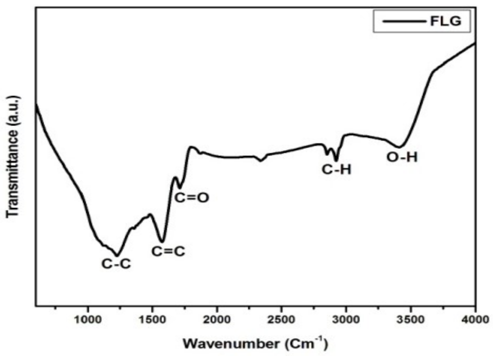

Figure 4. FT-IR spectra of FLG

FTIR spectrum of FLG materials for Figure 4. From the FT-IR spectra was observed the presence of functional groups in which the main absorption band at different carbon functionalities. The important characteristic peaks at 1229 cm^{-1} and 1580 cm^{-1} can be assigned to C–C stretching and C=C (aromatics) of carbon majority functional groups. The FT-IR peaks at 2921 cm^{-1} are assigned to the C–H stretching vibrations. The broad and wide peak at 3412 cm^{-1} is assigned to the O-H group stretching vibrations is significantly low band width due to directly exfoliation from graphite.

4. Electrochemical Measurements

The electrochemical performance of the Few Layer Graphene (FLG) was investigated by Cyclic Volta Metry (CV) measurements using three electrode system in 1Mole H_2SO_4 aqueous electrolyte. Figure 5 (a) shows the Cyclic Volta Metry (CV) curves at different scan rates (100 mV/s and 50 – 30 mV/s) in the potential range of -0.55 to 0.64 V vs. Ag/AgCl electrode.CV curves are observe that all represent similar shape with a pair rectangular shape, which can be attributed to the electrochemical double layer capacitor (EDLC) defect free surface FLG and in the interlayer structure pure carbon.

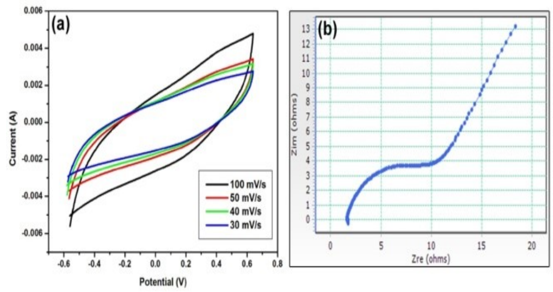

Figure 5. (a) Electrochemical analysis of CV for FLG and
(b) Impedance spectra of FLG

To electrochemical impedance spectroscopy (EIS) measurements were employed FLG material was obtained Nyquist plots shown in Figure 5 (b). The semicircle in the high frequency region reflects the charge transfer resistance (Rct). It indicates that Rct at the electrode/electrolyte interface the charge transfer resistance decreases in FLG material. From Nyquist plots Rct – 8.57 ohm was determined from the intercept of the semicircle on the real part axis at high frequency region. From EIS analysis FLG materials is low Rct value, electrode is obviously

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good conducting few layer materials that the energy storage applications.

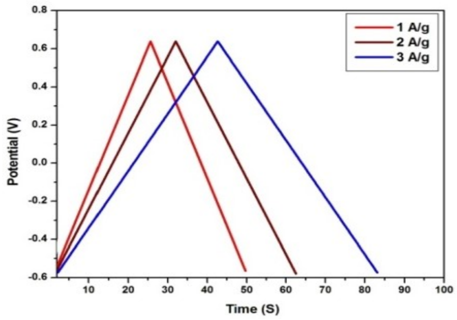

Figure 6. Charge–discharge curves atdifferent current densities 1-3 A/g.

The electrochemical analysis of galvanostatic charge–discharge was measured for Few Layer Graphene (FLG) material. The Specific capacitance (Csp) was calculated using following the equation (Eq - (1).

$$Csp = \frac{I \times \Delta t}{m \times \Delta V} \quad - \quad \text{Eq-(1)}$$

where Eq. (1) Csp (F/g) is the specific capacitance single electrode, I (A) is discharge current, (s) Δt (s) is the discharge time, ΔV represents the potential window during charge - discharge, and m (g) is the mass of the active material. Figure 6 From charge-discharge using Eq-(1) calculated the supercapacitors specific capacitances value 33.61 F/g, and 25.6 F/g and 20.28 F/g different current density values 1 A/g, 2 A/g, 3 A/g respectively. The FLG materials with promise that the excellent achievements for future energy storage device applications.

5. Conclusion

A facile, economic and practical method we have successfully demonstrated a facile scalable technique for producing relatively high-quality few layer graphene by kitchen blender method. The large scale produced FLG nanosheets with little nanometer thickness production of few layer graphene in average from SEM and HR-TEM. The XRD and Raman analysis were observed good crystalline and defects free graphene. The FLG materials were good conductive and free of surface plane defects and any other further oxidation. This was achieved using high Shear cleavage-exfoliations in bulk graphite to graphene mechanical-exfoliation through kitchen blender method. As prepared few layer graphene was done first time used as a electrode materials for supercapacitors applications. From electrochemical studies measurements CV is Electric Double Layer Capacitor (EDLC) nature. The galvanostatic charge-discharge was exhibited maximum specific capacitance Specific Capacitance of few layer graphene Amphere / gram(SCP) of FLG value of 33 F/g was calculations for current density at 1 A/g. The kitchen blender can strongly reveals that industrial scale exfoliation production or future energy applications.

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