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Original research article

Production of graphene powder by electrochemical exfoliation of graphite electrodes immersed in aqueous solution

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ABSTRACT

In our research, graphene powder was prepared by electrochemical exfoliation of graphite electrodes immersed in electrolyte solution of a sulfuric acid, nitric acid and distilled water ($H_2SO_4/HNO_3/H_2O$) with applied +10 V for 50 min. Graphene powder was characterized by XRD, Raman spectroscopy, AFM, SEM, and FTIR to investigate the structural, morphological, and chemical properties. The XRD analysis was showed polycrystalline structure of graphene with sharp peak at $2\theta = 26.61$ and broad peak at $2\theta = 54.68$ along (002) and (004) orientation respectively which was consistent with the interlayer spacing of normal graphite. Raman spectrum was demonstrated two intensive peaks at 1580 cm^{-1} and 1354 cm^{-1} for I_G and I_D confirmed with graphitic carbon-based materials. The AFM and SEM were exhibited the morphology of graphene powder has different shapes and sizes with a few agglomerates of crumpled and rippled structure. FTIR spectrum showed an absorption band at 1643.41 cm^{-1} assigned to the C=C stretching vibration of the hexagonal ring also there were oxygen-containing functional groups such as C—O and O—H.

1. Introduction

Graphene is a two-dimensional (2D) layer of carbon atoms ordered into a hexagonal structure called honeycomb lattice [1]. Graphene, one of the allotropes (carbon nanotube, fullerene, diamond) of elemental carbon [2], is a planar monolayer of carbon atoms with a carbon–carbon bond length of 0.142 nm [3]. Graphene exhibits superior electrical conductivity and a high charge carrier mobility (20 $m^2 V^{-1} s^{-1}$) [4] because electron tunneling occurs within its structure allowing electron movement at relativistic speeds. It has been reported to have the fastest electron and hole mobility than any other material [5]. It also has a high specific surface area (2630$m^2 g^{-1}$), which could be likened to that of a soccer pitch per gram) [6], excellent mechanical strength and stiffness, good elasticity, superior thermal conductivity, a broad electrochemical window and can offer as optical transparency [7]. A particularly promising graphene production technique is based on the obtainment of colloidal suspensions from graphite, or its derivatives [8]. In spite of other methods like epitaxial growth, chemical vapor deposition, and micromechanical exfoliation, this approach is both scalable, affording the possibility of high-volume production, and versatile in terms of chemical functionalization, which, on the other hand, is sometimes exploited to favor graphene obtainment and its dispersion [9]. The electrochemical approach has the advantages of being single-step, easy to operate, environmentally friendly (if using ionic liquid electrolytes or aqueous surfactants) and operates at ambient

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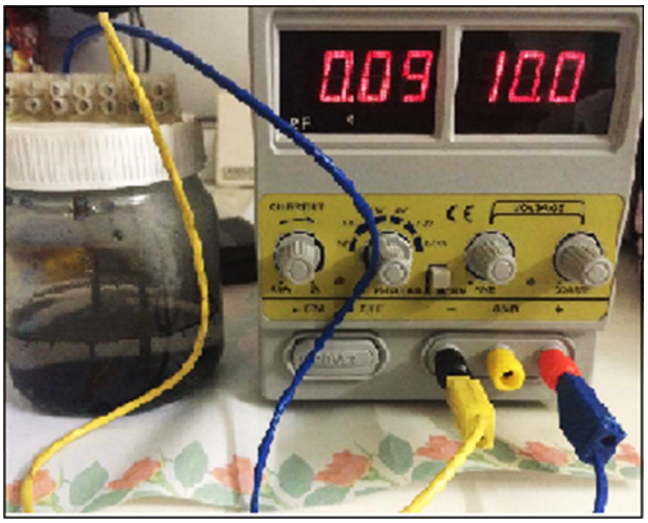

Fig. 1. Experimental setup of electrochemical exfoliation technique.

conditions [10]. Highly controllable flakes can be formed without the need for volatile solvents or reducing agents. The process can take several minutes to hours to complete and the reported results are encouraging for the fast-processing of large quantities of graphene flakes [11]. The electrochemical method utilizes a liquid solution (electrolyte) and an electrical current to drive structural expansion (oxidation or reduction), intercalation and exfoliation at a piece of graphite (rod, plate, wire) to produce graphene flakes. The experimental arrangement uses a monopolar, undivided electrolysis cell. The yield, productivity and properties of graphene flakes can be tuned by controlling the electrolysis parameters and electrolytes [12]. In this paper we attempted to prepare graphene by using graphite (pencil) as anode and cathode electrodes and immersed into the electrolyte solution (HNO_3,H_2SO_4 and H_2O), which will act as intercalate and studying the structural, morphological, and chemical properties.

2. Experimental details

Electrochemical exfoliation was carried out in an electrolysis cell shown as Fig. 1. This cell has graphite electrodes (pencil) as anode and as cathode and the separation distance between graphite electrodes was fixed at 4 cm shown as Fig. 2(a). These electrodes were immersed in electrolyte solution of sulfuric acid H_2SO_4 (0.69 gm) and nitric acid HNO_3 (0.19 gm) dissolved to 1000 ml of de-ionized water to make pH solution value around 3 at room temperature. The electrochemical exfoliation was done by first applying DC voltage of +1 V for 5 min and then we increased the voltages for 5 min +1 V until reached +10 V at 50 min. The application of high voltages on the anode resulted in the gradual exfoliation of graphite through edges. Afterwards, the graphene foam was extracted from electrolysis cell demonstrated in Fig. 2(d) and dried by using vacuum oven at 200 °C for two hours.

3. Material characterization

Structural properties were performed by X-ray Diffraction (XRD) according to the Joint Committee on Powder Diffraction Standards (JCPDS) card, using Shimadzu XRD-7000 X-ray diffractometer using CuKα (λ = 1.54050 A°) irradiation operated at 40 kV and 30 mA and by Raman spectrometer (Sentera Raman microscope Bruker co., Germany), which is double monochromatic instrument use a green laser (λ = 532 nm) as an excitation source. Morphological properties were characterized by scanning electron microscopy (SEM) (The VEGA Easy Probe) and atomic force microscopy (AFM) using a scanning probe microscopy (CSPM-5000) instrument. Chemical properties measured by Fourier transform infrared (FTIR) spectra with KBr disc were recorded using: FT-IR-8400S Shimadzu in the range of 400–4000 cm^{-1} at room temperature.

4. Results and discussion

4.1. Structural properties

Fig. 3 shows the XRD pattern of graphene powder prepared by electrochemical exfoliation method, the XRD exhibited the structure of graphene is polycrystalline has two peaks, a strong sharp diffraction peak at 2θ= 26.61 °, corresponds to an interlayer distance of 3.346 °A for (002) which is consistent with the interlayer spacing of normal graphite according to JCPDS card (230064), which has interlayer distance equal to 3.35 °A and a weak and broad diffraction peak at 54.68 ° of 2θ