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ORIGINAL ARTICLE

Exfoliation of graphene sheets via high energy wet milling of graphite in 2-ethylhexanol and kerosene

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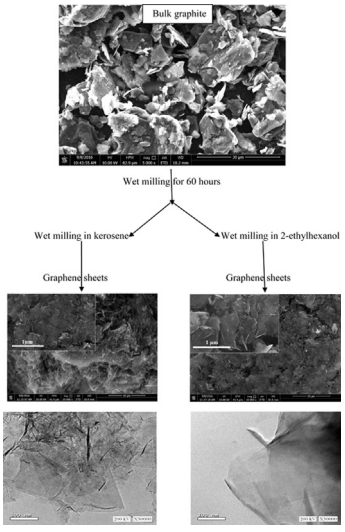
GRAPHICAL ABSTRACT



Abbreviations: XRD, X-ray diffraction; TEM, transmission electron microscopy; SEM, scanning electron microscopy; E-H, 2-ethylhexanol; K, kerosene.

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ABSTRACT

Graphene sheets have been exfoliated from bulk graphite using high energy wet milling in two different solvents that were 2-ethylhexanol and kerosene. The milling process was performed for 60 h using a planetary ball mill. Morphological characteristics were investigated using scanning electron microscope (SEM) and transmission electron microscope (TEM). On the other hand, the structural characterization was performed using X-ray diffraction technique (XRD) and Raman spectrometry. The exfoliated graphene sheets have represented good morphological and structural characteristics with a valuable amount of defects and a good graphitic structure. The graphene sheets exfoliated in the presence of 2-ethylhexanol have represented many layers, large crystal size and low level of defects, while the graphene sheets exfoliated in the presence of kerosene have represented fewer number of layers, smaller crystal size and higher level of defects.

Introduction

Graphene is known as an atomic layer of graphite, which is also the essential unit for fullerenes and CNTs. It is a two dimensional (2D) crystal that is stable under ambient conditions [1,2]. Single sheets of graphene are expected to have tensile modulus and eventual strength values like those of single wall carbon nanotubes (SWCNTs) and have a vast electrical conductivity. Similar to SWCNTs, graphene sheets serve as fillers for the improvement of electrical and mechanical properties in composite materials [3]. Graphene has exceptional in-plane structural, mechanical, thermal and electrical properties. These properties make it attractive for application in many research fields [4,5].

Defects in graphitic materials are important for enhancing the performance of carbon-based materials for practical applications. Because of the high anisotropy of the mechanical strength or the electrical conductivity between the in-plane and out-of-plane directions [6]. For example, to avoid the slip of the graphitic plane with respect to its neighbors, orientational disorder of the graphite planes is useful, and it is essential for enhancing the average isotropic mechanical strength. The different types of defects can be investigated by Raman spectroscopy [7–12]. Carbon allotropes show their fingerprints under Raman spectroscopy typically by D, G, and 2D peaks around 1350 cm^{-1}, 1580 cm^{-1} and 2700 cm^{-1} respectively due to the change in electron bands. Identification of these features allows characterization of graphene layers in terms of number of layers present [13]. The integrated intensity ratio I_D/I_G for the D band and G band is widely used for characterizing the defect quantity in graphitic materials [13]. Although there are different synthesis methods of graphene, they can be simply classified into two categories: top down approach and bottom up approach [1]. The most well-known of these methods are mechanical exfoliation [10], electrochemical exfoliation [14,15], chemical-derived [16], chemical vapor deposition (CVD) [17,18], epitaxial growth on SiC [19], and arc discharge [20]. Graphene can also be produced by unzipping CNTs with strong oxidizing agents, laser irradiation or plasma etching [21]. Intercalation compound methods have also been used to obtain graphene through spontaneous exfoliation of graphite [22].

Mechanical milling has been employed for producing graphene via different ways that include the production of colloidal dispersion of graphene in organic solvent [23], the synthesis of functionalized graphene nanoplatelets by mechanochemical milling [24], the production of graphene through ball milling of graphite with oxalic acid dihydrate [25], the synthesis of graphene nanosheets via ball milling of pristine graphite in the presence of dry ice [26], and the production of graphene by using ball milling of graphite with ammonia borane [27]. The main goal of the present research is to employ the wet milling in the presence of kerosene and 2-ethyl-hexanol separately to process and manipulate graphite powder for producing graphene sheets in the powder form with tunable characteristics.

Experimental

Wet milling process was performed using a planetary ball mill (PM.100 CM, from Retsch, Haan, Germany), Hardened steel vial (500 cc), Hardened steel balls (5 mm in diameter). Graphite powders (Sigma Aldrich, <20 μm, Schnelldorf, Germany) were milled at which the weight of the milled graphite powders was 10 g, and the weight of the milling balls was 500 g, then, the ball to powder ratio was 50:1 (i.e. $B/P = 50$). The milling speed was 400 rpm, and the milling time was 60 h. The graphite powders were milled in the presence of both kerosene (commercially available, from ExxonMobil company, Cairo, Egypt), and 2-ethylhexanol ($\geqslant$99.6%, Sigma Aldrich, Saint Louis, MO, USA). The prepared samples were centrifuged at 5000 rpm for 20 min to be separated from the solvent. Heat treatment of the prepared samples was performed in a tube furnace under the flow of argon gas for 3 h at 600 °C. Structural characterizations were performed via X-ray diffraction (XRD- PANalytical's X'Pert PRO diffractometer, Almelo, Netherlands), and Raman spectroscopy (Bruker Senterra instrument, Ettlingen, Germany, with a laser of 532 nm). On the other hand, Morphological characteristics of graphite powders and the prepared graphene sheets were investigated by scanning electron microscopy (Quanta FEG 250 (FEI, Hillsboro, USA), and Transmission electron microscopy (TEM-JOEL-JEM-2100, Tokyo, Japan).