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Synthesis and Characterization of Graphene sheets from graphite powder by using ball milling

Síntesis y caracterización de láminas de grafeno a partir de polvo de grafito mediante molienda de bolas

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Abstract

Due to the rising trend in 2-Dimensional material, graphene has gained a lot of interest in the recent past. Graphene is the 2D carbon allotrope with high strength and improved mechanical, chemical, and electrical properties. Despite being excellent properties among other types of carbon allotropes but still, graphene use is limited because of its costly synthesis technique. In this research, a cheap and effective method is adapted for the preparation of graphene from graphite powder. The graphite powder is thermally treated to prepare the exfoliated graphite then exfoliated graphite is milled to produce the 2D graphene sheets. The synthesized graphene is characterized by X-Ray Diffractometry (XRD) and Scanning Electron Microscope (SEM). The XRD results show that graphene is successfully synthesized, and SEM results show that graphene is 2D which can be used in various applications. This research provides a direction for the synthesis of graphene from graphite powder on an industrial scale.

Keywords: Graphene; Graphite; Ball Milling; Synthesis; Characterization.

Resumen

Debido a la tendencia al alza en el material bidimensional, el grafeno ha ganado mucho interés en el pasado reciente. El grafeno es el alótropo de carbono 2D con propiedades mecánicas, químicas y eléctricas mejoradas de alta resistencia. A pesar de tener excelentes propiedades entre otros tipos de alótropos de carbono, el uso del grafeno es limitado debido a su costosa técnica de síntesis. En esta investigación se adapta un método económico y efectivo para la preparación de grafeno a partir de polvo de grafito. El polvo de grafito se trata térmicamente para preparar el grafito exfoliado y luego se muele para producir las láminas de grafeno 2D. El grafeno sintetizado se caracteriza por difractometría de rayos X (XRD) y microscopio electrónico de barrido (SEM). Los resultados de XRD muestran que el grafeno se

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sintetiza con éxito y los resultados de SEM muestran que el grafeno es 2D que se puede usar en varias aplicaciones. Esta investigación proporciona una dirección para la síntesis de grafeno a partir de polvo de grafito a escala industrial.

Palabras clave: grafeno; grafito; molienda de bolas; síntesis; caracterización.

1. Introduction

Graphene can easily be regarded as a 'Material of the Future' after all the research being conducted on its different properties and applications in multiple areas like energy storage devices such as capacitors and transistors. Graphene is a single layer of carbon atoms packed into a two-dimensional honeycomb-shaped lattice. It has received immense attention since its discovery in 2004 [1] due to its exceptional desired properties such as outstanding electron mobility up to 230,000 cm^2/Vs [2], [3], 3000 W/m.K thermal conductivity which is on par with Carbon nanotubes (CNTs) and diamond and 12 times as compared to Copper [4], [5], [6]. 2.3% absorption of visible light [7], the strong mechanical strength of 130 GPa [8], and a high specific surface area of 2630 m^2/g [9], [10]. It has a density lower than 1 g/cm^3 with an ultra-large surface-to-volume ratio. These properties can be altered depending on any of the many synthesis techniques to synthesize graphene.

High chemical stability and electrochemical activity make graphene an active non-metal electro-catalyst or catalyst support for energy storage systems. Out of many potential applications of graphene, their use as protective films or as additives in coatings for anti-corrosion purposes has seen an increased level of interest in the last decade [11].

The Graphene synthesis method plays a vital role in the outcome of its structure and properties. Moreover, doping graphene with different active component leads to outstanding performances in some applications, and studies has been conducted in this area too. Synthesis of graphene may be conducted through one out of two broad approaches. 'Top-down approach' or 'bottom-up approach'(Figure 1).

Top-down production of graphene often involves the reduction or exfoliation of powdered graphite into graphene [12]. The most prominent top-down methods are mechanical exfoliation, LPE, electrochemical exfoliation, and chemical oxidation–reduction [13]. Bottom-up synthesis of graphene involves the use of hydrocarbon compounds as precursors and building upon them in such a way that we end up getting graphene [30]. Some of the most widely used bottom-up approaches include chemical vapor deposition (CVD) which gives a very high yield but is just not viable for high-scale production, thermal pyrolysis, epitaxial growth, laser-assisted synthesis, and organic synthesis [7], [14], [15], [16], [17], [18], [19].

2. Methodology

2.1. Materials and Chemicals

All the chemicals including Graphite powder, tri-sodium acetate, ethanol, and acetone are purchased from Sigma Aldrich.

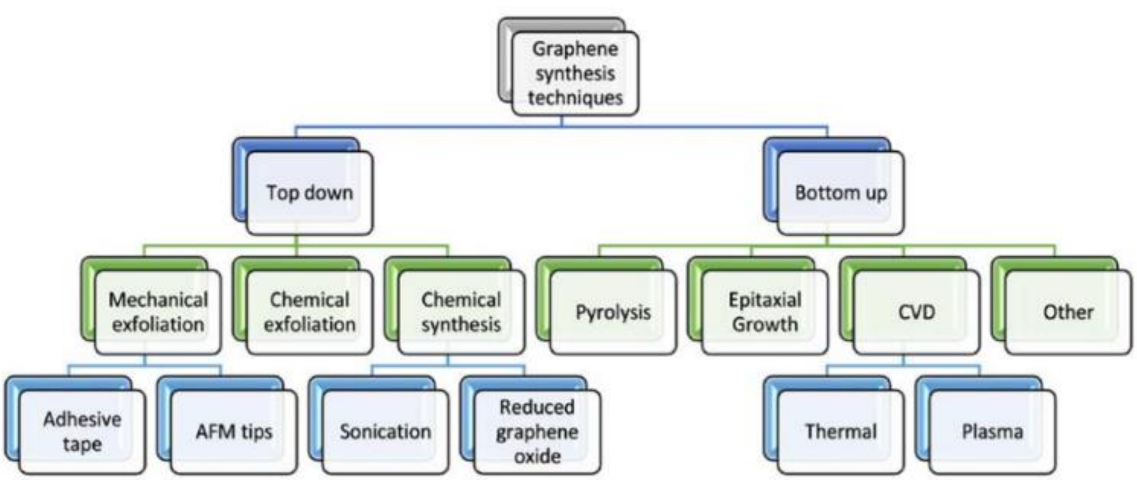

Figure 1. Graphene Synthesis flow chart [20].

2.2. Synthesis of Graphene

First graphite powder is thermally treated by keeping it in the furnace for 3 hours at 500º C. This is done to expand the graphite. Then the expanded graphite is dispersed in ethanol to prepare the graphitic solution. The solution is prepared in the presence of tri-sodium acetate to avoid agglomeration. The solution is then placed in ultrasonic bath at 80º C for 1 hour for exfoliation. After an ultra-sonification, the solution is dried by keeping it in the oven for 4 hours at 90º C.

The exfoliated graphite is dispersed in acetone and then wet milling is performed by using a ball mill equipped with hardened steel balls. The milling is performed at 400 rpm for 4 hours. The mild speed is selected to ensure that sheer forces are present to exfoliate the graphene sheets. The black dispersion is placed for centrifuge at 1000 rpm to remove the thicker flakes. Figure 2 shows the schematic view for the preparation of graphene from graphite powder.

2.3. Characterization

The prepared graphene is characterized by XRD to ensure that graphene is successfully synthesized and to determine the morphology of synthesized graphene SEM is conducted.

3. Results and discussions

The process for the synthesis is conducted in a controlled environment to ensure that exfoliation is carried out properly. The milling speed is taken to be mild because it is producing the sheer forces which are enough to prepare the 2D graphene sheets. The centrifuge is conducted because the mixture contains both graphene sheets and the residue. The graphene sheets are lighter when it is placed for centrifuge, graphene sheets come at the upper side and residue goes to the bottom side when can be separated by a simple pouring method.

To ensure that graphene is successfully synthesized and to determine the crystalline behavior of synthesized graphene XRD is conducted by Bruker axS Germany. The XRD pattern of the synthesized graphene is shown in Figure 3 which is in good agreement with the published literature [22]. The sharp peak observed at $2\theta = 26.4^{o}$ and the small peaks observed at 44.1º and 56.3º are showing the presence of carbon within the graphene sheets and is showing that graphene is successfully prepared as confirmed by the literature [22].

The morphology and shape of the prepared graphene are determined by Scanning Electron Microscopy (SEM), Joel Japan. The SEM analysis is conducted by passing the electron beam at 20 kV to determine the shape of the prepared graphene. Figure 4 shows the SEM analyses of the graphene sheets taken at the magnification of 4000X. Both Figures 4(a) and 4(b) are showing that 2D graphene sheets are successfully synthesized with an average size of the graphene flakes is around 20 µm by using the method discussed in the methodology section. The 2D graphene sheets can use a nano-filler in improving the mechanical properties, electrical properties, optical properties, and chemical properties of any base material. This graphene is also able to use as cathode material in metal-air batteries [23].

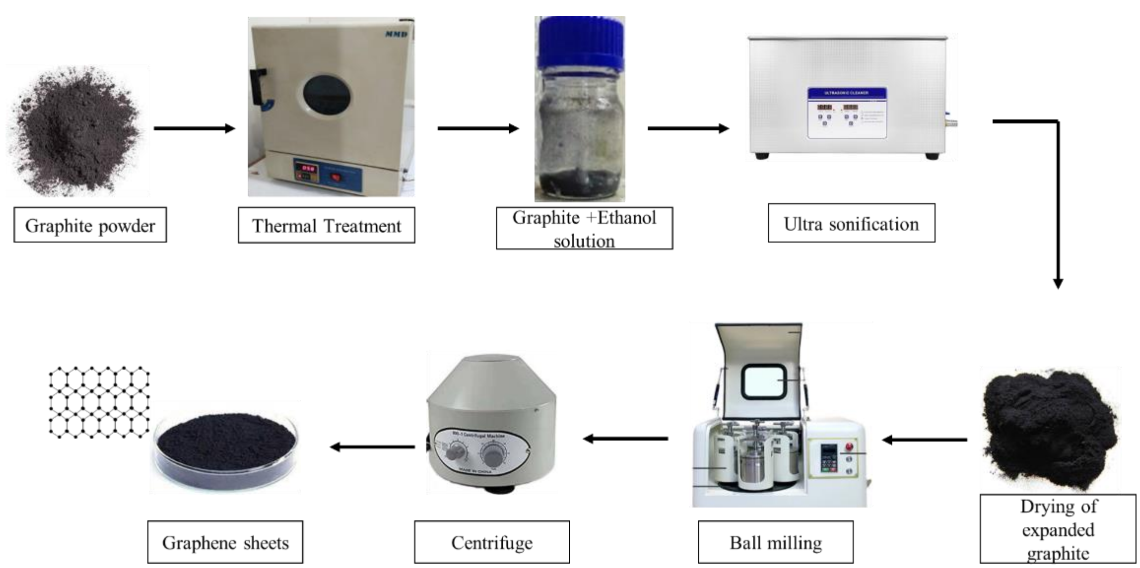

Figure 2. Schematic view for graphene preparation.

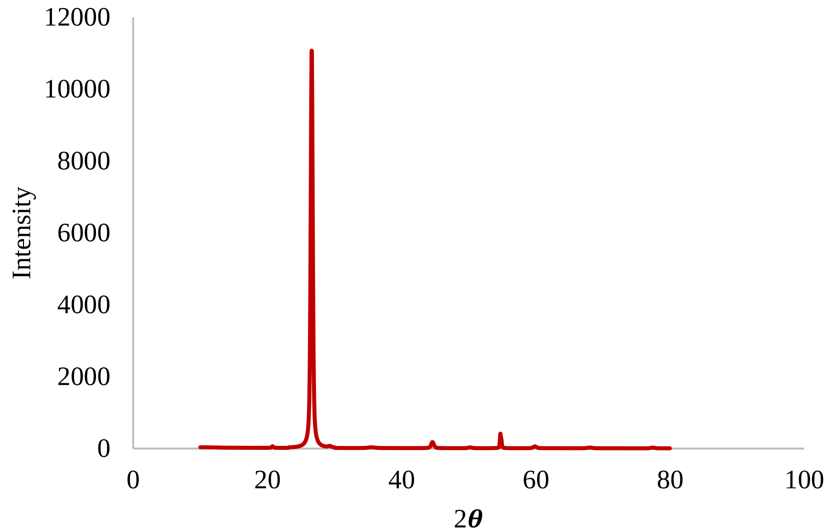

Figure 3. XRD pattern of synthesized graphene.

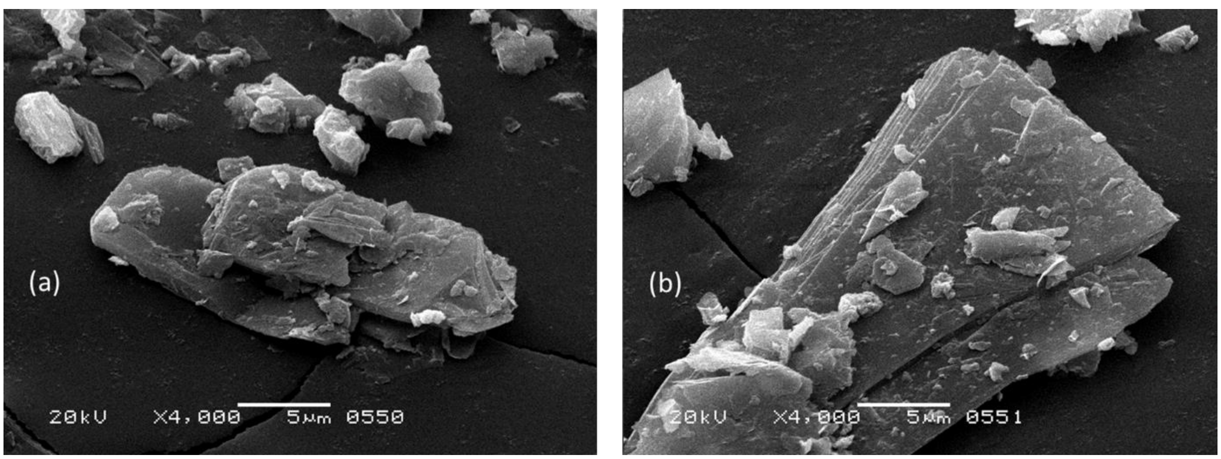

Figure 4. SEM images for the prepared graphene.

4. Conclusions

In this research, graphene is first synthesized by using a ball mill and is then characterized by XRD and SEM. Graphene is successfully synthesized from graphite powder. First graphite powder is thermally treated to prepare the exfoliated graphite, then exfoliated graphite is then balled and milled to form the 2D graphene sheets. The mild speed of 400 rpm for ball milling is selected to ensure that there are enough sheer forces to transform the exfoliated graphite into 2D graphene sheets. The graphene sheets are separated from residue via centrifuge which works on the separation of heavy and light particles by centrifugal forces. The prepared graphene is characterized by XRD and SEM. The XRD results show that graphene is successfully synthesized as the results are in good agreement with the literature. The SEM results show that the shape of synthesized graphene is 2 dimensional which can be good to enhance the chemical, electrical, thermal, and mechanical properties. The average size of the prepared graphene flakes is found to be around 20 µm.

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