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## Synthesis and characterization of Graphene Oxide in suspension and powder forms by chemical exfoliation method

### ABSTRACT

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In this study, an efficient and facile technique for preparing graphene oxide in suspension and powder forms was presented based on a modification on Hummers' method followed by an additional ultrasonic process. The method involved the provision of graphene oxide from graphite by reaction of potassium permanganate and sulfuric acid with stabilizing the medium complex. Furthermore, this study evaluated the functionality of graphene oxide in powder form in comparison with suspension using Fourier transform infrared spectroscopy. In addition, prepared graphene oxide powder was characterized using X-ray diffraction method for investigating the exfoliation of graphite to graphene nanosheets. Moreover, transmission electron microscopy and atomic force microscopy techniques were employed to demonstrate the structure of resulting graphene oxide in suspension form. The presence of graphene oxide nanosheets including an average thickness of about 2-4 nm was proved via microscopy observation of the prepared nanosheets.

**Keywords:** *Chemical synthesis; Graphene Oxide; Graphite nanosheet; Oxidation; Chemical exfoliation.*

### INTRODUCTION

Recently graphene as a new nano filler has attracted appreciable attention for many applications. This increased interest is due to proper properties of graphene such as high thermal and electrical conductivity, high Young's modulus, effective mechanical properties and high tensile strength [1-3]. These outstanding properties have made graphene a superior candidate for many applications in automotive, aerospace, composite, and electronic industries [4, 5]. Graphene is a two-dimensional structure of sp<sup>2</sup>-bonded carbon sheet which is densely packed in a honeycomb lattice. Before the discovery of graphene in 2004, it was believed that the thermodynamic fluctuations are not consistent with the existence of two-dimensional crystals at the finite temperature [6].

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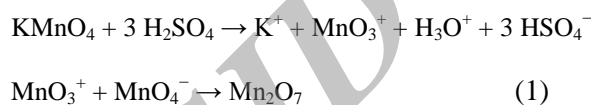
However, successful synthesis of this individual structure by Novoselov and Geim attracted a considerable attention for this new class of nano-fillers [7].

There are different methods for preparing graphene, which main are mainly different in the thickness of the prepared graphene, and the volume of provided product. Some methods lead to a good degree of platelet exfoliation and some others are more efficient for large-scale manufacturing. High-yield production methods for graphene preparation are mainly desirable for some applications such as composite materials production and conductive films manufacturing [8]. In general, four categories of methods can be introduced for graphene production. The first category is chemical vapor deposition and thermal-induced exfoliation [9-12]. The second is micromechanical exfoliation of graphite, which is also known as the 'scotch tape' or 'peel-off' method [13, 14]. The third is direct synthesis, such as epitaxial growth, and bottom-up organic synthesis; and finally the fourth category is the chemical exfoliation of graphite and solution-based reduction of graphene oxide (GO) [15-18].

It seems that using of graphite oxide as the foundation of graphene preparation is the most efficient method amongst all mentioned methods for large-scale graphene production [7, 17, 18]. Graphite oxide formation involves the reaction of graphite with the strong oxidizers such as sulfuric acid, nitric acid, potassium chlorate, and potassium permanganate. The presence of oxygen-containing functional groups such as hydroxyl, carboxyl, carbonyl and epoxide results in increasing *d*-spacing of graphite oxide. Besides, the presence of oxygen-containing functional groups leads to the hybridization of oxidized carbon atoms from planar  $sp^2$  to tetrahedral  $sp^3$  [11]. Graphite oxide is strongly hydrophilic and because of the existence of functional groups attached on the basal or edge of sheets can easily exfoliate in water. Therefore, the ultrasonic process might leads to the formation of a partially stable dispersion of GO in water. However, GO is an insulating material and thus it cannot be used without further reduction as a conductive nano-filler.

There are different methods introduced for chemical preparation of GO by the oxygenic treatment of graphite via one of the three well-known methods developed by Brodie, Staudenmeier, and Hummers [19-21]. The

produced graphite oxide by these methods still remains a layered structure, but it includes much lighter color compared to graphite due to the loss of electronic conjugations [3]. In particular, the Hummers' method involves oxidation process of potassium permanganate and sulfuric acid. In this method the active species is diamanganese heptoxide ( $Mn_2O_7$ ) which is obtained via the reaction of monometallic tetraoxide and  $MnO_3^+$  (Eq. 1). This dark oil is far more reactive than its monometallic tetraoxide counterpart as placed in contact with organic compounds or heated up to temperatures greater than 55 °C [22].



Predicted procedure for oxidation of graphite in present of  $Mn_2O_7$  is depicted in Figure 1 as a general schematic of the oxidation reaction. Due to the strong bonding of water molecules and the basal plane of GO after ultrasonic process, the interlayer spacing of graphite sheets increases noticeably [22]. These strong bonds form through hydrogen bonding of water molecules with the oxygen in the epoxide groups of the GO. These interactions are the main reason for high stability of GO in water in comparison with other common solvents [22]. Therefore, oxidation of graphite followed by sonication in water will lead to a highly exfoliated GO in partially stable form.

An efficient technique for the preparing GO by a modified process based on Hummers' method was presented here (see Figure 1). This study employed a technique based on the oxidation of graphite followed by sonication of the expanded GO in water. The method was resulted to a partially stable suspension of graphene oxide via a secure, fast, facile and low-cost technique. In addition, a fully efficient and simplistic process was reported for preparing GO powder with a proper range of nanosheet thicknesses. Also the prepared graphene oxide solution and GO nanosheets powder were characterized employing Fourier transform infrared (FT-IR) spectroscopy, atomic force microscopy (AFM), transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques.

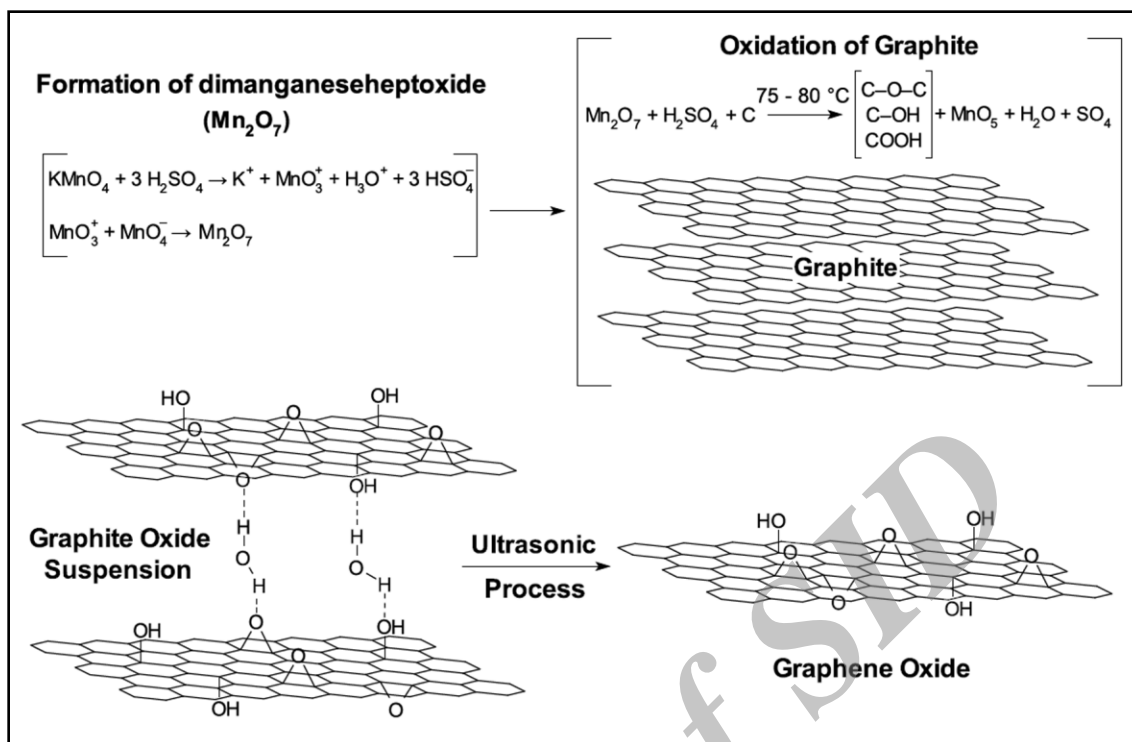


Fig. 1. Procedures used in this study for preparation of graphene oxide

## EXPERIMENTAL

### Chemicals and Materials

Graphite powder was purchased from LECO Co. and potassium permanganate ( $KMnO_4$ ), sulfuric acid ( $H_2SO_4$ ) and all other reagents and solvents were provided from Merck Chemicals Co.

### Preparation of Graphite Oxide

Natural graphite powder was used for preparing graphene oxide by a modification in Hummers and Offeman's method [21]. 1 g of graphite powder was put into a round bottom flask. Thereafter, 100 ml of sulfuric acid (98%) was added into the flask. Afterward, the mixture was stirred and gradually 6 g of  $KMnO_4$  was added to the mixture. Once more the mixture was stirred by a magnet stirrer at room temperature for 2 h. The mixture was subsequently allowed to react for 4 h at 75-80 °C. Then, 300 ml of distilled water was added to the reaction mixture. Followed by 15 min, the reaction was terminated by adding 50 ml of  $H_2O_2$  aqueous solution (25 ml, 30%), resulting in a yellow-brown mixture (see the supplementary information given in Figure 1(a-d)). Finally, the

mixture was centrifuged and washed out for five times with a 10% HCl solution and was repeatedly washed out with water till the neutral pH of the filtrate was reached.

### Preparation of Graphene Oxide suspension and Graphene Oxide powder

Graphite oxide was dispersed in water (5 mg/ml) and sonicated (20 kHz, 1000 W, 80% power) for 20 min. In order to remove un-exfoliated GO particles, the mixture was centrifuged for 10 min at 5000 rpm to obtain the graphene oxide suspension. Graphene oxide suspension was gradually sprayed out on a glass surface kept at 110 °C and then was separated layer by layer from the surface to achieve graphene oxide nanosheets powder. The procedure of graphene oxide powder preparation was naive and efficient and led to multilayer graphene oxide powders formation. Hydrazine hydrate at 3:1 ratio was used to reduce GO suspension to graphene. This reduction was conducted at 90 °C for an hour. The graphene suspension was centrifuged to remove any unstable particles and was dried out at 100 °C for an additional 24 h to remove all the residual solvent.

### Characterization Methods

AFM were provided using a Dualscope DS 95-200, DME. Samples for AFM were developed on a freshly cleaved mica surface. Roughness parameters were determined using a Femtoscanner image processing software (version 2.3.94). Chemical characteristics of graphene oxide were characterized by Nicolet IR100 FT-IR spectrometer. SEM images were obtained employing a Hitachi S4160 scanning electron microscope. XRD patterns were presented on an Inel Equinox 3000 diffractometer ( $\text{Cu } k_{\alpha}$  ( $\lambda = 1.54056 \text{ \AA}$ ) at 40 kV and 30 mA). Hielscher UIP1000hd homogenizer (20 kHz, 1000 W) was used for the ultrasonic process. TEM investigations were performed using an instrument of PHILIPS CM120 model.

## RESULTS AND DISCUSSION

### Chemical characterizations

FTIR technique was employed for chemical analysis of graphene oxide in both suspension and powder forms as shown in Figure 2. The effects of powdering process on functionality of nanosheets was investigated by analysis the FTIR behavior of nanosheets prepared through powdering process compared to FTIR spectra of GO suspension dried overnight and during 3 days at 80 °C in a vacuum oven. Four intensive peaks were appeared on the spectrum of overnight dried GO at wave numbers including: ~629, 1639, 2091 and 3400  $\text{cm}^{-1}$  (Figure 2a). Appeared intense peaks at ~1639 and 3400  $\text{cm}^{-1}$  were corresponding to C=C stretching vibrations of graphene and hydroxyl (-OH) symmetrical stretching vibrations of the water, respectively [2, 23]. Moreover, the vibration peak at wave number of around 2091  $\text{cm}^{-1}$  was contributed to C-H band stretching. In addition, there is no sign of the epoxide and carboxyl groups in the spectrum of overnight dried GO suspension (see Figure 2a). This behavior might be related the high degree of residual moisture of GO according to the high intensity of hydroxyl vibrations.

FTIR spectrum of GO suspension dried during 3 days at 80 °C in a vacuum oven showed no sign of residual moisture in the structure of GO sheets (see Figure 2b). The FTIR spectrum of dried GO showed two peaks around 586 and 863  $\text{cm}^{-1}$

mainly were corresponding to C-H deformation vibration. The absorption peaks at 1007 and 1137  $\text{cm}^{-1}$  were contributed to C-O-C bond stretching in the structure of epoxide group [23, 24]. Moreover, the peak around 1570 and 3400  $\text{cm}^{-1}$  were corresponding to C-OH stretching vibrations in hydroxyl groups and C=C stretching vibrations, respectively [24, 25]. Furthermore, vibration peaks at 1675 and 2900  $\text{cm}^{-1}$  were corresponding to C=O and C-H stretching vibrations, respectively [23, 26]. In addition, a peak appeared around 2360  $\text{cm}^{-1}$  was contributed to C-H deformation vibration.

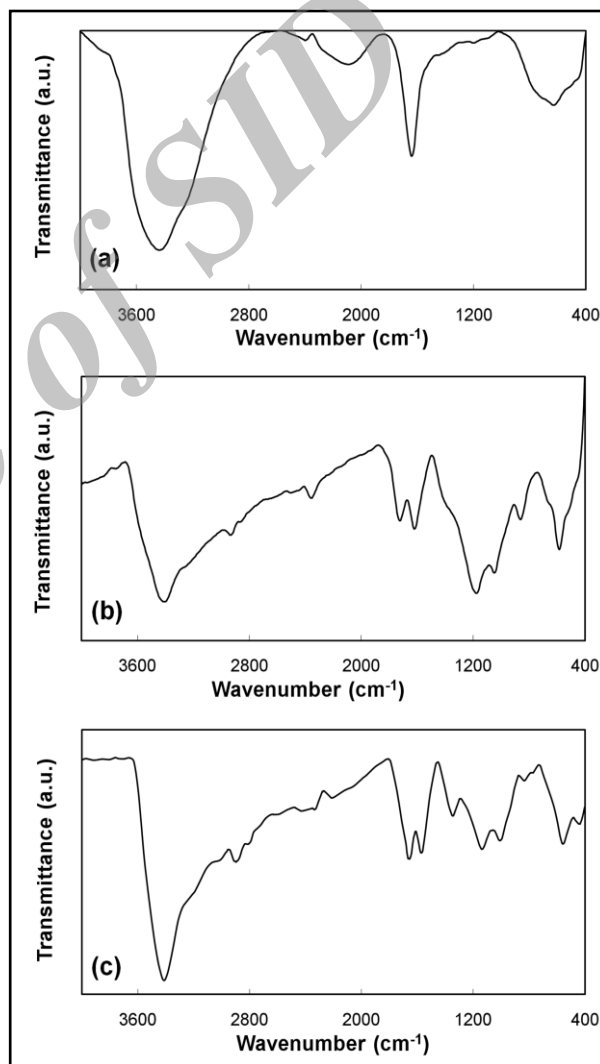


Fig. 2. FTIR spectra of overnight dried graphene oxide (a), graphene oxide dried through 3 days (b) and Graphene oxide powder prepared via powdering method (c)

The FTIR spectrum of GO powder was used to investigate the efficiency of the presented powdering process (Figure 2c). The spectrum GO powder showed two peaks around 450 and 560  $\text{cm}^{-1}$  mainly were corresponded to C–H deformation vibrations. However, the peaks obtained in the spectra of 3-days dried GO and GO prepared via powdering method were closely in the same patterns. Therefore, the presence of these vibration peaks verified the successful attachment of functional groups (hydroxyl, epoxide and carboxyl) on graphite surface during powdering process. The FTIR spectrum of 3-days dried GO showed no sign of residual moisture in comparison with overnight dried GO. Such behavior proves that 3 days drying of GO is enough for preparation of GO powder using vacuum oven. However with the use of presented powdering process this time period decreased noticeably. This fact clearly proves the full efficiency of GO powdering process in comparison with vacuum drying.

### Structural behavior

The structural behavior of original graphite, GO powder and reduced graphene were characterized using XRD method, as shown in Figure 3. A sharp reflection at  $2\theta=26.4^\circ$  in XRD pattern of the pristine graphite originates from the interlayer (0 0 2) spacing of  $d=0.34$  nm [27]. Upon oxidation, adjacent layers of graphite became about 0.7 nm apart ( $2\theta=11^\circ$ ) because of the intercalation obtained by oxygen groups. Such  $d$ -spacing is significantly larger than that of pristine graphite interlayer spacing ( $\sim 0.34$  nm). In addition, the reflection peak appeared around  $43^\circ$  in XRD pattern of GO, indicates that GO exhibits turbostratic disorder [28]. Moreover, after sonication, the significant peak at  $26.4^\circ$  approximately disappeared. This fact clearly proves complete exfoliation of graphite sheets to individual or few-layered graphene [9].

Individual GO sheets are expected to be thicker than individual pristine graphene sheets for the presence of oxygen-containing functional groups attached on both sides of the graphene sheet and the atomic scale roughness. This atomic roughness arises from structural defects ( $sp^3$  bonding) generated through oxidation on the originally flat graphene sheets. Therefore, because of the disorder structure introduced by reduction process, XRD pattern of the reduced graphene

oxide showed broadening behavior in the intensity of the peak at  $43^\circ$  besides shifting in the location of peak to lower angles. This behavior clearly proved full exfoliation of graphene and graphene oxide powders. Besides, high degree of exfoliation in XRD pattern of GO can be a clear evidence for high efficiency of powdering process.

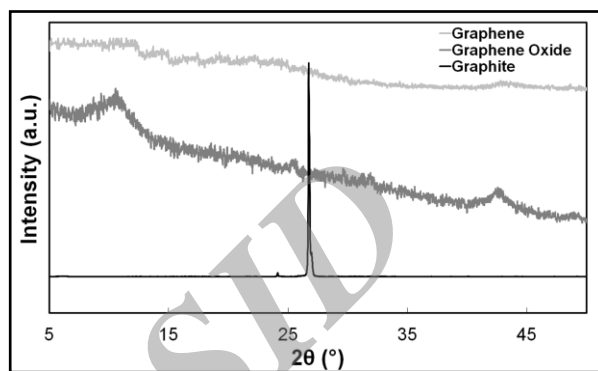


Fig. 3. XRD patterns of pristine graphite, graphene oxide and graphene

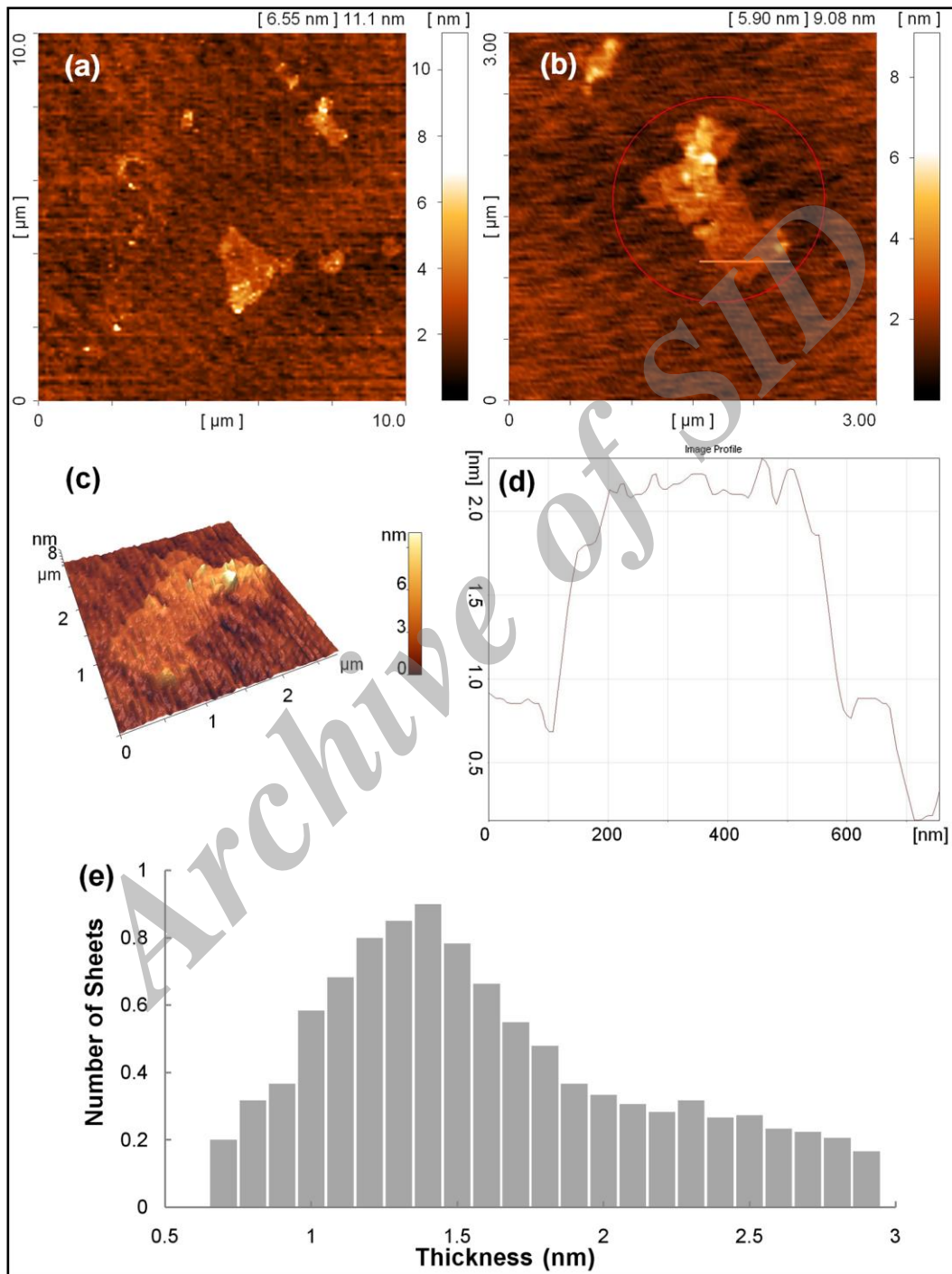
### Thickness measurements

AFM was employed to characterize the degree of exfoliation and thickness distribution of individual and few-layered GO sheets, as shown in Figure 4 and Figure 5. An individual GO sheet was selected (Figure 4a) for further investigation using the three-dimensional (Figure 4b) and profile views (Figure 4c) of GO. The thickness dispersion of individual GO sheet in the thickness size histogram (Figure 4d) revealed that the thickness of formed nanoplatelet was about 1–2 nm. The profile view (Figure 5b) and thickness size histogram (Figure 5c) of few-layered GO sheets revealed that the thickness distributions of nanosheets in bulk was in the average range of 2.5–4 nm. Generally, this range of thickness was responsible for expounding the height distributions of prepared GO in the average range of 1–4 nm. This range of thicknesses was reported as the proper range of thickness for single and multi-layered graphene in literature [8–10].

The values of mean roughness ( $R_a$ ) which shows the arithmetic average of the absolute values of the surface height and root mean square (RMS) roughness parameter ( $R_q$ ), as the standard deviation of feature height within a sheet were measured about 0.75 and 0.98 nm, respectively. Although the reported value for RMS roughness of GO film in

literatures was about 1.2 nm [29]. The oxidation process increased the roughness of graphite surface according to AFM 3D view of GO nanosheets and recorded roughness values. During oxidation of graphite because of cooperative binding of  $Mn_2O_7$

and graphite, linear clusters of epoxy groups are formed which lead to a bent sheet [30]. This curved structure leads to an increase in the amounts of  $R_q$  relating to the surface roughness.



**Fig. 4.** AFM images of graphene oxide nanosheet: Phase and topography (a), 3D view (b), Profile of GO nanosheet (c) and Histogram of thickness (d)

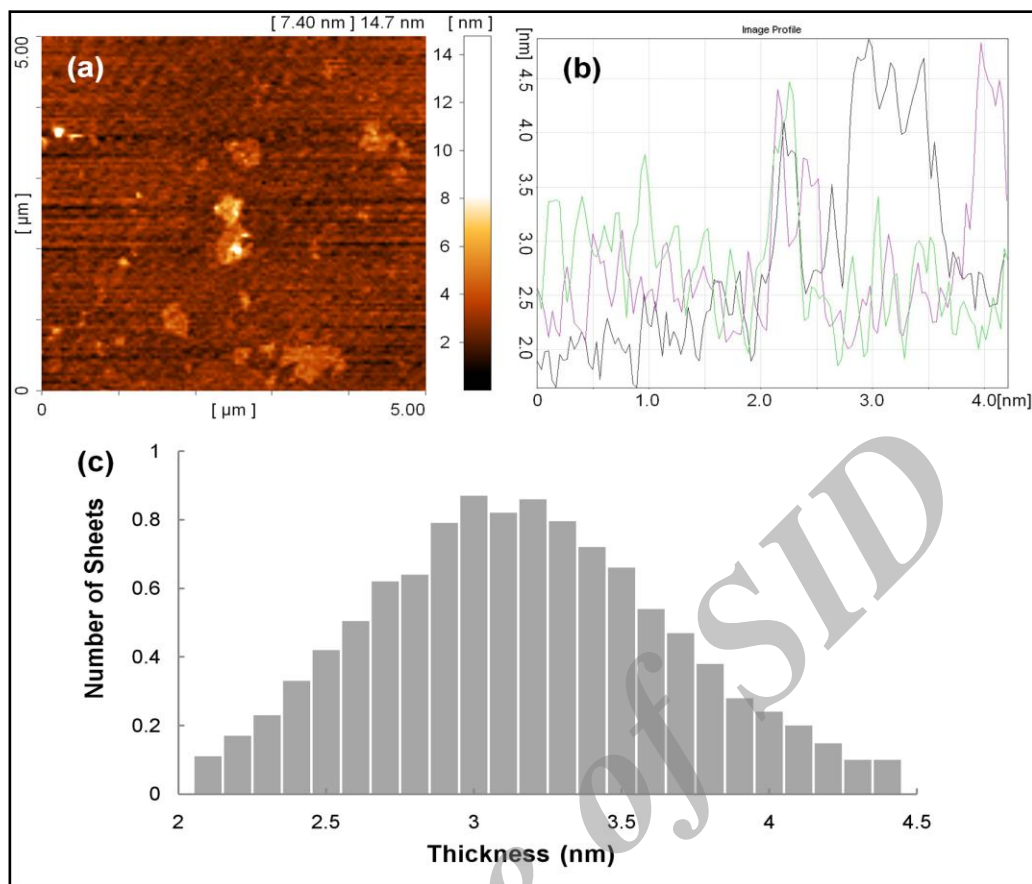


Fig. 5. AFM images of multilayered GO topography (a), Profile (b) and Histograms of thickness (c)

### Morphological studies

In addition to XRD, TEM analysis was conducted for further structural characterization of GO sheets in the suspension form, as shown in Figure 6. As depicted, graphene nanosheets were rippled and entangled with each other. In addition, TEM images of the nanosheets manifested the presence of monolayer-like graphene nanosheets in the bulk of prepared suspension (shown by arrow on Figure 6). According to the reported results for individual and few-layered GO nanosheets, the thickness of nanosheets were in the range of 2-4 nanometres which is in accordance with TEM images of the nanosheets (see Figure 5). In addition, corrugation and scrolling were observed for the GO nanosheets that this behavior is intrinsic to the nature of GO. This behavior is due to the thermodynamic stability of the two-dimensional GO structure. In addition such behavior results from microscopic crumpling of GO sheets that previously reported in literatures as well [7].

The morphology of reduced graphene, the overnight dried GO and GO powder were studied using FESEM method, as shown in Figure 7. Reduced graphene FESEM image (Figure 7(a)) revealed that the reduced GO material consists of randomly aggregated and wrinkled sheets that were closely associated to each other. These wrinkled sheets formed a disordered solid with fully exfoliated sheets. The overnight dried GO sheet (Figure 7(b)) presented a number of tiny wrinkles over the whole surface of the sheet with mountainous peaks. Moreover, SEM micrographs of GO powder (Figure 7(c and d)) displayed that through powdering process the GO sheets was completely torn into sheets with a range of thicknesses between 30 to 70 nm. This range of thicknesses represented a large aspect ratio for the GO nanosheets and came as a proof for the presentation of multi-layered GO nanosheets in a proper range of thicknesses in powder form.

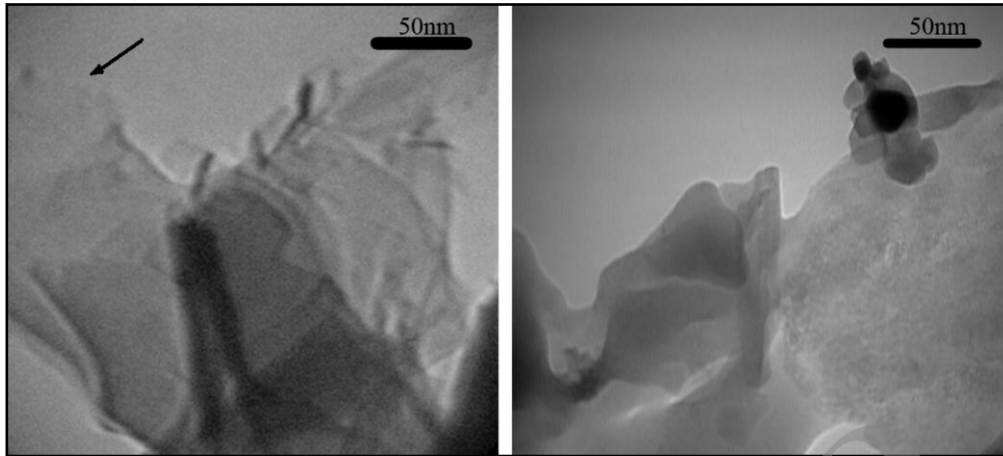


Fig. 6. TEM images of graphene oxide suspension

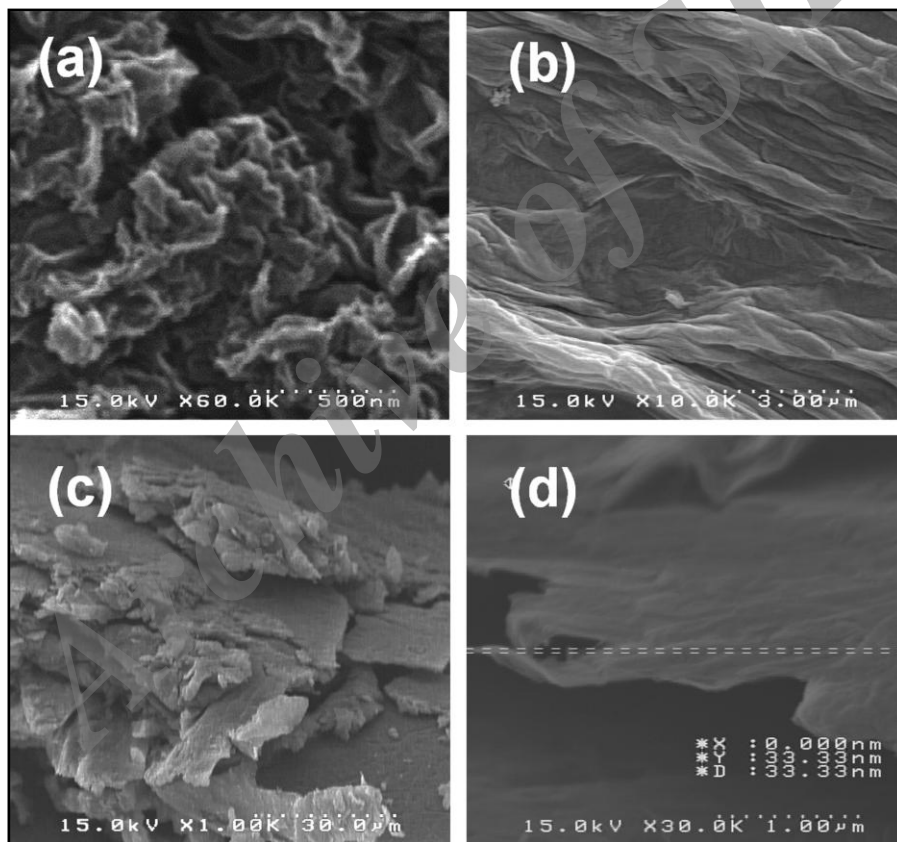


Fig. 7. SEM images of graphene (a), oven dried GO sheet (b) and GO powder (c,d)

## CONCLUSIONS

The profusion of natural graphite sources made graphene a more attractive candidate for new applications as nano filler. A facile, fast, efficient

and secure method for preparing a partially stable suspension of graphene oxide and graphite oxide nanosheets powder was presented in this study. Moreover, in this study the graphite powders were exfoliated into graphene oxide nanosheets using a



modified method based on Hummers' method followed by ultrasonic process. Beyond, a new trend for individual graphene oxide nano platelet's development was proposed and the final characteristics were confirmed through the characterization of surface chemistry, structural properties, thickness measurements, as well as the morphological observations. The key factor in decreasing the production time in reported method was to control the diamanganese heptoxide complex which is the main oxidizer through Hummers' oxidation process.

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