Journal of Ovonic Research Vol. 17, No. 3, May - June 2021, p. 253 - 259

Modification of hummers presses for synthesis graphene oxide nano-sheets and graphene oxide /Ag nanocomposites

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Graphite is a based on three-domination functionalized carbon content made up of millions of graphene layers. In this study, graphene oxide (GO) and graphene oxide\Ag nanocomposites (GO-Ag) have been synthesized in a single layer using a modified Hummer method. The morphology and structural characteristics of GO and GO-Ag nanocomposites were investigated by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), transmitted electron microscopy (TEM), and Raman spectroscopy. The results of TEM indicated the single and double layer structure with thickness of 4-6 nm for GO Nano-sheets prepared in this analysis. The particles size of silver on a surface of GO was to be less than 20 nm.

(Received March 4, 2021; Accepted May 12, 2021)

Keywords: Graphene oxide, GO\Ag, Hummers presses, Raman spectroscopy

1. Introduction

The global synthesis of nanostructure is due to numerous exciting and specific characteristics that promote its use in completely different fields such as, nano-diagnosis, nanomedicine, and antimicrobial products on the one hand and photocatalytic luminescence and photodiode reaction on the other, In particular graphene oxide and applications [1-5]..Graphene is a graphite-separable 2D product. Graphene is a composite substance that contains Sp2-hybridized carbon atoms [6,7]. Due to their ideal properties in different applications [8] graphene (G) -based materials have been extensively studied in recent years. The applications include membrane division [9,10], heavy metal [11,12], dye adsorption [12], microwave absorption [13,14], photocatalysis [15,16] and sensor applications [17,18]. This is the most important application of this application: One of them is GO, that can be synthesized from graphite flakes[19,20], a widely studied graphite-based materials. The oxygen content of graphene oxide plates in functional groups is capable of inducing hydrophilicity, and so it is informal for graphene oxide to alter the surface relative to others. Some methods could be used to produce the graphene oxide (GO). In 1859, the Brodie process was described for the first time, in which fuming Nitric acid (HNO3) and Potassium chlorate (KClO3) are being used as successful and highly and oxidants, respectively [21-25]. However, some weaknesses of this approach have been recorded.

The process takes about four days and needs Four cycles of oxidation, washing and drying to finish the reaction [19]. In addition, during the response stage, highly explosive (ClO2) gas can be generated by the mixture of chlorate and acid [26]. The Hummers Method, which was introduced in 1958, is currently the most frequently used method for graphene oxide synthesis. Sulfuric acid is used to oxidize graphite laterally with sodium and potassium permanganate. In order to complete the reaction within several hours, the hummer process uses potassium permanganate (KMnO4) to suppress difficulties in connection with the Brodie technique [27,28]. Moreover, chlorate does not minimize the risk of creating ClO2 gas in the reaction. It is suggested that the main role of sodium nitrate could enable the intercalation of sulfuric acid (H2SO4) which would be advantageous to graphite oxidation by oxidants, attractive considering the low oxidation potential of sodium nitrate (NaNO3).[29,30]. Sodium nitrate (NaNO3) acts as a graphite intercalant until the formation of graphite oxide (GO). The most important source of graphite to

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produce GO appears to be flake graphite [31]. Flake graphite structure had several faults that could be used as oxidation sites. However it is difficult to determine the graphene Oxide formation mechanism because of its complex structure and critical defects [32]. The majority of research focuses on the transition from C = C to C-O. However, the impact of structural changes in graphite flakes or diffusion in the oxidant has not been recognized in the most recent studies. Actually there are multiple graphite layers in the original graphite source. The oxidant must therefore first enter the middle layer before the reaction to oxidation can take place [33-35]. As a consequence, graphite oxidation also depends on the particle size of the original graphite, and the literature has shown that graphite oxidation increases when the particle size decreases and the oxidation time depends primarily on the particle size of its initial graphite[36]. Reducing the particle size (small graphite particles are perfect for preparing large-scale graph oxides) reduces the oxidation time [37-39]. The two-step method that is used to produce graphene out of graphite in the GO synthesis unit is known as oxidation of graphite to make graphene oxide, and washing and purification of graphene oxide from unwanted compounds (manganese salts, acids ,etc.) [40-43]. The modified Hummer method was developed from the graphene oxide sheets in this study. Graphene oxide/Ag nanocomposites have also been prepared and XRD, FESEM, TEM and Raman spectroscopy have been verified in previous preparations.

2. Experimental procedure

2.1. Synthesis of graphene oxide by modified hummer method

The graphene oxides nano-sheets were synthesized by the modified Hammer method. Accordingly, 2 gm of graphite powder and 0.5g of sodium nitrate ($NaNO_3$) was mixed with 50 ml of Sulfuric acid (H_2SO_4 98%). the mixture was placed in the ice bath to remained the temperature below 5°C and kept stirring for 30 min. Next, 5 g of potassium permanganate ($KMnO_4$) was added slowly to the mixture. After 15 h, 500 ml of distilled water was added under intense stirring. Then, 10 ml of 32% hydrogen peroxide (H_2O_2) was added to the mixture. A multi-step purification procedure was applied in order to isolate the reaction product. It was centrifuged, cleaned, and submerged in deionized water and a HCl (20) % solution for several steps. In the end, the black-brawn product was dried at room temperature over-night to obtain graphene oxide Nano-Sheets [44,45].

2.2. Synthesis of graphene oxide /Ag nanocomposites

1 gm of GO nano-sheets was dispersed in 40 ml of de-ionized water. then, 20 ml of silver nitrate (0.5 N) was added with an ice bath. The mixture continued to stir for 30 minutes, after which it was slowly added 25 ml, 1 N of sodium citrate with kept stirring for 6 hours. The brawn powder was separated and washed with de-ionized water for several times and dry it at room temperature to obtain GO /Ag nanocomposites as shown Fig. 1.[46,47].

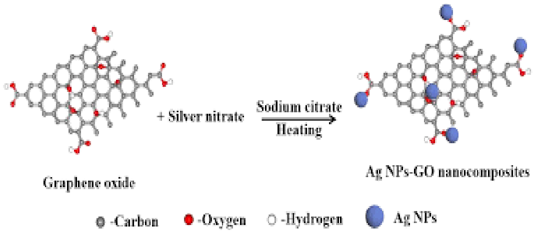


Fig. 1. Synthesis of GO /Ag nanocomposites [47].

2.3. Characterization of GO sheets and GO-Ag nanocomposites

Some techniques have been used to classify the prepared GO and GO/Ag. TEM model Jeol JSM-6010LV observed sample morphologies with 500 X and 60 kX magnification with a

5kV accelerating voltation. Before TEM analyzes, the samples (GO and GO/Ag powder) were discrete in the deionized and sonic water for approximately 15 minutes. SEM modell Jeol JSM-6010LV A total of 20 μL was lowered over a grid of 300-mesh Cu and dried at room temperature. Model D-5000 X-ray diffraction (XRD) was used to investigate the composition of the specimens by using Cu-Kα radiation (λ=0.154nm) source in θ/2θ. The XRD The XRD (10˚ to 80˚) and measurment temperature (25 °C) are 2 for each of GO &GO/Ag NPS. A Raman spectrometer by Thermo Scientific fitted with a 532-nm laser and a 10-inch target was taken at room temperature[44-48].

3. Result and discussion

3.1. FE-SEM spectroscopy

Detailed morphological analyses were carried out using a scanning electron microscope. The GO nano-sheet and GO-Ag nano-powder FESEM pictures appear in Figs. 2 and 3. The figures show nano-sheets with their typical wrinkles and folds. This finding verified that the GO exfoliation can generate two dimensional nano-sheets. Various layers of graphene oxide are observed in Figure 2, while AgNPs are deposed on the graphene oxide surface in Fig. 3.

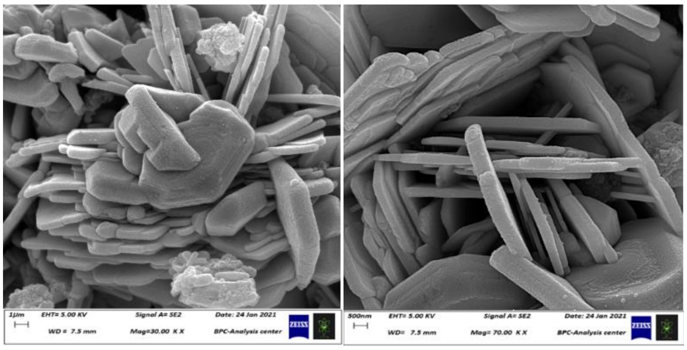


Fig 2. SEM images of GO nano-sheets.

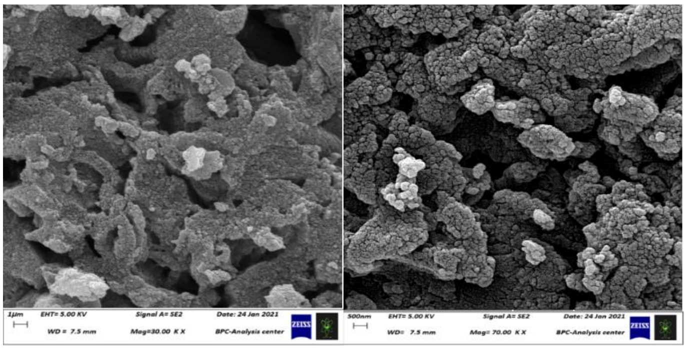


Fig. 3. SEM images of GO\Ag nanocomposite.

3.2. TEM spectroscopy

The TEM images show a sheet with different fonts of the region examined, which shows different thicknesses (Figure 4 and 5). Dark regions represent the dense stacking nanostructures of several layers of GO and GO\Ag with certain feature groups of oxygen. The higher levels of clarity indicate much thinner films of a few layers of graphene oxide \silver resulting from stacking exfoliation. GO and GO\Ag Significantly wider surface area with high delaminated graph layers transparency (about one-layer thickness). The TEM images indicates that GO sheet layers are 4 to 6 nm thicker than graphs of a single layer. The increased thickness is due to the insertion of functional groups containing oxygen that make them thicker and alter the GO sheet surface (figure 4). The particles size of silver on a surface of GO was to be less than 20 nm. The diffusion rate of Ag nanoparticles growth could rely heavily on the number of Ag atoms given to standing clusters (figure 5). Finally, autocatalytic oxidation comprises the repeat of steps 1 and 2 to absorb remaining AgNO3 to obtain the nanoparticles of Final Ag.

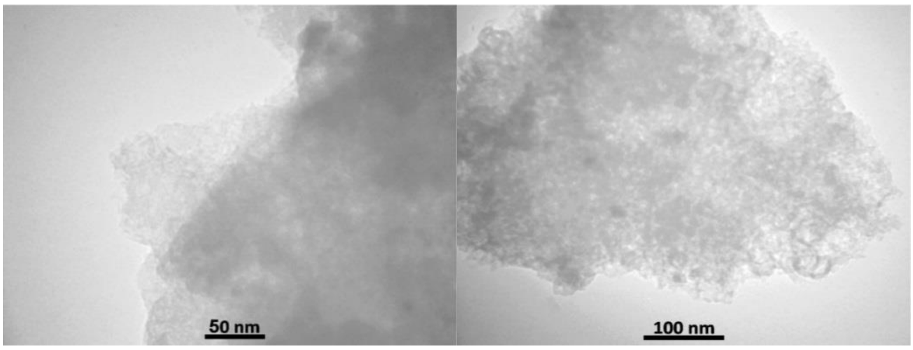


Fig.4. TEM images of GO nano-sheets.

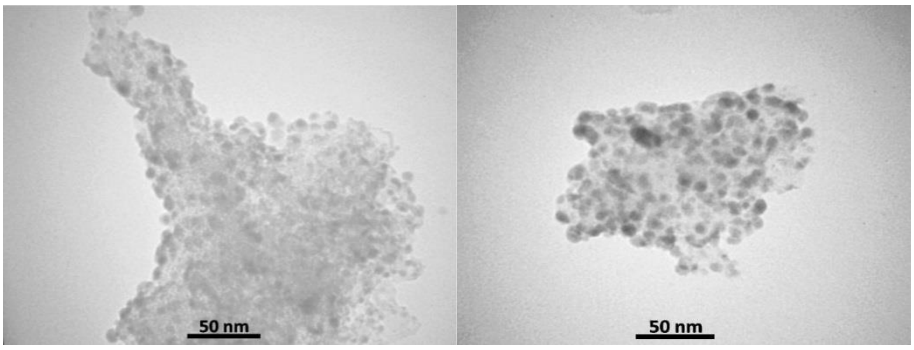
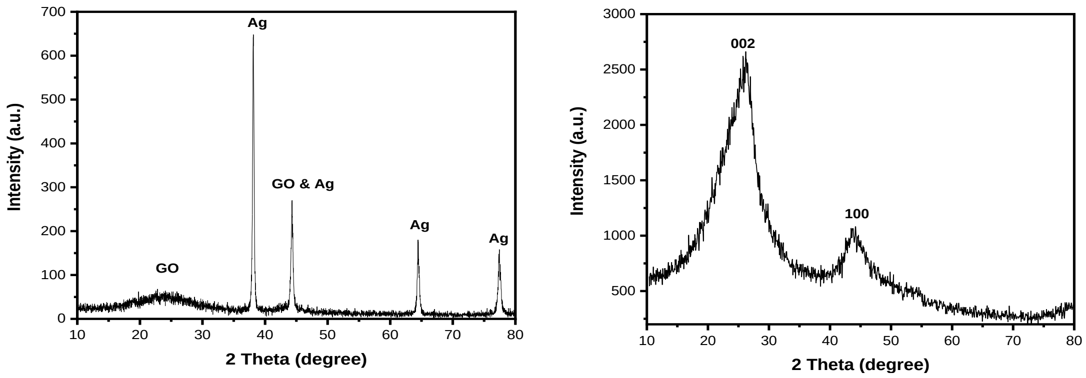


Fig.5. TEM images of GO\Ag nanocomposite.

3.3. XRD

The XRD pattern of Graphene oxide Nano-sheets and Graphene oxide -Ag nanocomposites are shown in Fig. 6 (a and b). GO XRD pattern indicates a 2θ = 10.19° value that fits the plane (100). A small peak of 2θ =20.93 °, representing the plane (002) was also observed. In Graphene oxide Nano-sheets, the spacing in the layer increases in comparison to graphite because of the inclusion of functional groups containing oxygen during graphite oxidation. A peak related to both Graphene oxide Nano-sheets and Ag nanoparticles was seen on the Graphene oxide Nano-sheets -Ag nanocomposite (interlayer spacing of 1.392 nm). But a small adjustment of peak to lower 2θ confirms that AgNPs are intercalated with GO sheets.

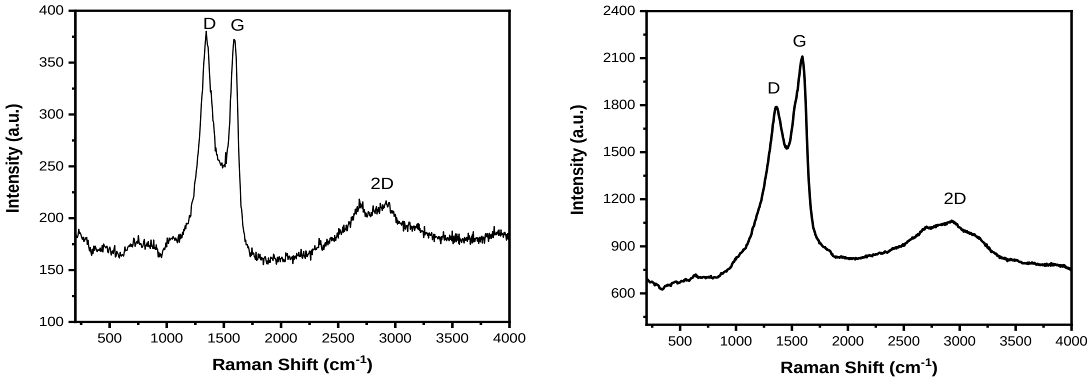


(a) *(b)*

Fig.6. XRD of a) GO Nano-sheets and, b) GO-Ag nanocomposites.

3.4. Raman spectroscopy

Fig. 7 (a and b) shows the Raman spectra of the prepared GO sheets and GO/Ag nanocomposites samples. The Raman spectra of the prepared GO sheets showed typical (a) and GO-Ag nanocomposites (b) peaks near (1355 and 1592 cm−1), (1345 and 1572 cm-1) respectively. The Raman's shift to the sp2 region of GO sheets and GO-Ag nanocomposites. A more clearly realized 2D splitting phenomenon is the luxurious spectrum of Fig. 7 (a and b), in which the existence of the 2D band is visible. In comparison, the 2D band of GO plates and GO-Ag nanocomposites are more symmetric, showing that their crystallites have decreased the number of graphic layers. However, the 2D range of GO-Ag and GO-Ag represents a sharp and symmetrical summit.



(a) *(b)*

Fig.7. Raman spectra of ***a)*** *GO nano-sheets and,* ***b)*** *GO-Ag nanocomposites.*

4. Conclusion

In conclusion of this work applied modified hummer's method as to produce graphene oxide (GO) sheets. The different conventional hummer method synthesis something to get monolayer graphene oxide and graphene oxide -sliver nanocomposites (GO/Ag) to increase the physical and chemical properties of graphene oxide When the silver nanoparticles are scattered on a surface. The GO sheet layers are 4 to 6 nm thicker than graphs in a single layer shown in the images of TEM. The increased thickness is due to functional oxygen grouping added which makes the surface of the GO sheet thicker and changed. Finally, Ag NPs was to be less than 20 nm in a GO surface.

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