One-Step Synthesis and Characterization of Reduced Graphene Oxide/Silver Nanoparticles Using Chemical Reaction

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Abstract. The rGO-Ag NPs nanocomposite was successfully synthesized by one-step with a chemical reduction method. This method's characteristics by three points are easy, short time does not exceed three mint and one-step. The rGO-AgNPs nanocomposite was analyzed with a (UV-VIS) spectroscopy. Fourier Infrared Spectrometer (FTIR), X-ray Diffraction, Atomic force microscopy (AFM), scanning electron microscope (SEM), Energy Dispersive X-ray Spectroscopy EDX. The results have shown the graphene oxide has been reduced as confirmed by UV-VIS, FTIR, and XRD, and the AFM, SEM analysis confirmed the presence of silver particles in nanoscale and anchored on the reduced graphene oxide sheets with high density and homogenous.

Keywords: rGO-AgNPs, UV-VIS, FTIR, XRD, AFM, SEM, X-ray Spectroscopy.

1. Introduction
Nanomaterials have gained great importance in recent decades due to their distinctive properties such as (shape, size distribution, morphology, chemical composition, and crystal environment). Researchers are interested in synthesizing nanomaterials with different methods, for different materials, and for different applications [1].

One of these nanomaterials is graphene that interest due to its excellent properties such as large surface area, high chemical stability, maximum mechanical strength, and unique electronic and thermal conductivity these properties arise from the flat single layer of carbon atoms encased in a two-dimensional lattice-shaped like a honeycomb [2,3,4].

The nanocomposites enhancement of the properties of the nanomaterials, such as graphene oxide/silver nanoparticles GO/Ag NPs, GO/Co, GO/FeO₃ and etc. Graphene papers provide a new path for material development as a substrate for nanoparticles, Moreover, graphene can be an effective support material to disperse nanoparticles and prevent their agglomeration, among these nanoparticles, the Ag NPs have attracted increased attention due to their optical, electronic, and anti-bacterial properties [5,6,7]. The GO/Ag NPs nanocomposite has an interesting structure, making it promising for various applications, for example as an anti-microbial and anti-bacterial agent [8].

Most importantly, the volume and loading of Ag NPs can be controlled on graphene sheets, which are critical for the design and application of these nanocomposites. There are several
methods to synthesize GO/Ag NPs, such as physical, hydrothermal, sol-gel, and chemical reaction\[9\]. On the other hand, the researchers looking for methods, easy, simple, not expensive, short time and given best results.

The chemical reaction will be used because it can be controlled the particle size, homogenize distribution, low cost, and a short time to prepare the nanocomposite. But, the reports that using this method to synthesize the rGO-Ag NPs nanocomposite have used somewhat expensive materials, special laboratory conditions, and a long time, and the reaction process was more than one step \[10,11,12,13\]. The proposed method in this study will be easy, one-step, inexpensive, short time, and did not require special laboratory conditions.

2. Materials and Methods

Silver nitrate (AgNO3), Sodium Borohydride (NaBH4) was purchased from market commercial, and Graphene oxide has been synthesized by the Hummer method as in previous work \[14, 15\].

Method of synthesis of silver nanoparticles with graphene oxide

The rGO-Ag NPs have prepared by adding 10 mg from GO to 10 ml distilled water to be the concentration 1mg/ml gradually added to 0.3 mM of AgNO3 aqueous solution under vigorous stirred at room temperature followed by the slowly gradual addition of 0.1M of NaBH4 to the mixture solution, the color of the mixture turns from light yellow to dark yellow, then purple, and then dark brown. Then the rGO-Ag NPs nanocomposites were obtained by centrifugation at 8000 rpm, washed with distilled water to get rid of impurities and reduce toxicity. Finally, the rGO-Ag NPs is ready for analysis.

On the other hand, silver nanoparticles were prepared without graphene oxide, mix 5 mL from 0.3 mM of AgNO3 and 5 mL from 0.1M of NaBH4, in the same way.

3. Results and Discussion

To study the optical properties of rGO-Ag NPs and Ag NPs are checked used UV-Visible Spectrophotometer, the results are shown in Figure 1.

![Figure (1)](image)

**Figure (1)** Show the Uv-vis absorption for AgNPs and rGO-Ag NPs.

Figure (1A) showed a curve for Ag NPs only and appeared one peak at (392 nm) refers to the silver nanoparticles Ag NPs, and the figure (1B) for rGO-Ag NPs shown two peaks, one at

\[392 \text{ nm}\]
(400 nm) refers to Ag NPs and second at (268 nm) refers to reduced graphene oxide represented the transitions between $\pi = \pi^*$ in $C = C$ bound, the two peaks have redshift that indicates the nanoparticles of silver seemed to be getting smaller and the graphene oxide has been reduced, this indicates the removal of the oxygen groups and the close proximity to graphene.

The curve in figure (1b) indicated that the silver nanoparticles have the anchor on the reduced graphene sheets. This result compatible with other reports [16].

For knowing the present of the oxygen functional group in graphene oxide, FT-IR transmittance spectra in figure(2) shown the reduction process is mainly the removal of oxygen functionalities with partial restoration of aromatic graphene network (sp2) as show in peaks at 3259.60 cm$^{-1}$ and 1637.05 cm$^{-1}$ which is related to O-H bonds and second peak was a carboxylate C = C bonds, the O-H bonds maybe came from water because the rGO-Ag NPs dissolved in water when taking the result, and the carboxy bonds appears only without any groups, that means the graphene oxide has been reduced by removing the oxygen groups. This result compatible with reports [17].

![Figure (2) Show Transmission FTIR spectra of rGO-Ag NPs.](image)

The XRD analysis is further used to confirm the formation of the rGO-Ag NPs nanocomposites. As shown in Figure 3, the XRD peaks at $2\theta = 38^\circ$, $45^\circ$, and $64^\circ$, respectively, are attributed to the (111), (200) and (220) crystallographic planes of face-centered cubic (fcc) Ag. These results correspond to the International Card for silver nanoparticles as in fig (4) and with other reports [18].

![Figure (3) Show The XRD analysis.](image)
Additionally, in fig (3) the presence of small peaks at 2θ = (34, 61) due to the presence of silver oxide according to the International Card for silver oxide nanoparticles as in fig (5).

Moreover, there are two peaks at 2θ = (12) for the crystal plane (001) returns to GO, and the peak at 2θ = 26 for the crystal plane (002) returns to rGO, this confirms that the graphene has been reduced. Also, this result corresponding with other reports [19].

The crystal particle size of Ag nanoparticles in the nanocomposite can be calculated by Scherer’s equation as in equation (1) [20] and the results as shown in Table (1).

\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \]  

(1)

where is \( D \) the average grain size, \( \lambda \) is the X-ray wavelength, \( \beta \) is full width at half maximum (FWHM) and \( \theta \) is the diffraction angle of the diffraction peak profile.

| No. | 2θ  | d (nm) | hkl | β   | C.S (nm) |
|-----|-----|--------|-----|-----|----------|
| 1   | 38.260 | 0.23505 | 111 | 0.221 | 35.92    |
| 2   | 44.479 | 0.20352 | 200 | 0.255 | 32.25    |
| 3   | 64.577 | 0.14420 | 220 | 0.239 | 33.82    |

From table (2) that the average cystatin size is 33.996 nm and it is within the nanoscale and compatible with the results of UV-Vis.
To analyze the surface morphology rGO-Ag NPs, two techniques were used, the first was (AFM) and the second was (SEM). Where were the results of the AFM images in figure (6) showing sample structure in 2D and 3D and statistical distribution, and in figure(6c) the histogram shows that the average grain size of the rGO-g NPs is (71.26 nm) and this indicates that the material is within the nanoscale as in the results for the previous analyses.

![AFM images of rGO-Ag NPs](image)

**Figure (6)** shows AFM images of the rGO-Ag NPs.

The morphology of rGO-Ag NPs shown in figure (7), the images show that the reduced graphene oxide was wrinkled and the silver nanoparticles have homogeneous distribution over rGO sheets.

Moreover, all the silver nanoparticles Ag NPs are spherical, and from the figure (7 B) the aggregation of Ag NPs is absent, approximately. The size for silver nanoparticles on average diameters is less than 30 nm.
Additionally, the elements for the rGO-Ag NPs were detected by EDX analysis, as in figure (8) that show several peaks, and the atomic and weight percentages for these elements as shown in table 2.

By observing table (2), the ratios of carbon, oxygen, and silver are 79.47 %, also the carbon ratio more than the oxygen ratio indicated that the graphene oxide has been reduced. Furthermore, the ratio of silver has represented that the silver nanoparticles are decorated with the reduced graphene oxide sheets with high density.

Figure (7) SEM for rGO-Ag NPs.

Figure (8) EDX spectrum for rGO-Ag NPs.
Table (2) Show the atomic and weight percentage for the sample rGO-Ag NPs nanocomposite.

| Elements | W%  | A%  |
|----------|-----|-----|
| C        | 27.50 | 47.92 |
| Ag       | 24.46 | 4.75  |
| O        | 20.49 | 26.81 |
| Si       | 12.42 | 9.26  |
| Na       | 6.79  | 6.18  |
| S        | 3.65  | 2.38  |
| Cl       | 3.43  | 2.03  |
| K        | 1.26  | 0.67  |
| Total    | 100   | 100   |

Indeed, the silicon is present in a clear percentage, and this is due to deposition rGO-Ag NPs on the silicon wafer, as well as the emergence of elements (Na, S, Cl, K) and are considered as impurities came from the preparation process of graphene oxide as well as from the preparation process of rGO-Ag NPs nanocomposite and these ratios of these elements are very small compared with carbon, oxygen, and silver.

4. Conclusions

The study proved that the proposed method was easy, one-step, inexpensive, and a short time to synthesize RGO-Ag NPs. Additionally, the results, shown that the graphene oxide has been reduced and at the same time successfully, the silver nanoparticles anchored on the reduced graphene oxide sheets. Moreover, the grain and crystalline size of the silver nanoparticles are in the nanoscale, as well, the shape of the AgNPs is spherical, and the nanoparticles are dispersed evenly distributing on the reduced graphene oxide sheets. This nanocomposite with these good specifications can be applied in several different applications in all areas of life.

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