Effect of method used on the morphology of AgO NPs

Hiba Shihab Ahmed¹ and Haider Nazar Hussein²

¹University of Diyala Presidency
²The General Directorate for Education of Diyala

Abstract: This article describes the preparation of silver oxide nanoparticles (AgO NPs). Natural sources as a green method were used compare with chemical in procedure. Then the properties of the products were compared to each other by Ultraviolet-visible, X-Ray diffraction, Field Emission Scanning Electron Microscopes and Transmission electron microscopy. The silver oxide nanoparticles were prepared using environmentally friendly manner using AgCl₂ salt as a source of silver metal with the help three aqueous extracts of castor leaves as a reducing factor and NaOH solution as a precipitating agent. Silver oxide nanoparticles were characterized by UV, XRD, FE-SEM and TEM techniques. UV-vis examination exhibited that the size of AgO NPs was 138 and 134 nm for chemical and eco-friendly method, respectively. X-ray diffraction (XRD) examination showed that the size of silver oxide nanoparticles was 23 and 17 nm for chemical and eco-friendly method, respectively. FE-SEM and TEM images gave different sizes and shapes of silver oxide nanoparticles.

Keywords: Silver oxide nanoparticles; eco-friendly and chemical methods.

INTRODUCTION

Nanomaterials and nanotechnology have gained great importance in modern research. Nanomaterials defined as physical substances with one dimension at the smallest between (1 to 100 nm), have attracted a lot of attention due to their unique features that set them apart from their bulk counterparts (Harish, Ansari, et al., 2022). The study of nanomaterials adopts a materials science-based approach to nanotechnology, making use of developments in materials synthesis and metrology that were made to assist micro-fabrication research (Jeevanandam et al., 2018). From chemical nature materials the nanoparticles can be synthesized, the most common being metals and metal oxide (Harish, Tewari, et al., 2022). The several different morphologies nanomaterials exist such as spheres, cylinders, platelets and tube etc. (Istiqola & Syafiuuddin, 2020). (Ali et al., 2023) One of substances used in nano formulation is silver and silver oxide nanoparticles (Ealia & Saravanakumar, 2017). Silver nanoparticles has antimicrobial properties, that lead to use in filters to purify drinking water and removal any other environmental pollutants. (Bhardwaj et al., 2021) Silver and silver oxide nanoparticles has a strong antimicrobial activity, this is the major direction for development of nano silver products, so it is one of the promising products in drugs industry. (Bruna et al., 2021) The variety and multiplicity of silver nanoparticles synthesis methods is important in nanotechnology research. (Nguyen et al., 2023)

On the other hand, in recent years many approaches been successfully developed, such as eco-friendly, chemical and physical for metal and metal oxide nanoparticles synthesized, eco-friendlly approach that mean plant extract used (all part of plant such as leaves, flowers, seeds, barks and roots ) for reducing silver ions to silver nanoparticle, additionally, it work capping agents in Ag-NPs preparation. (Singh et al., 2018), (Kuppusamy et al., 2016) Aslam and co-workers, studied the Ag-NPs synthesis use S. procumbens plant aqueous extract, this process was successfully and confirmed properties by different techniques. (Aslam et al., 2021) Aala and co-workers, studied the AgO NPs synthesis by chemical method via different salt and applied it cytotoxicity against MCF-7 breast cancer cell line. (E Sultan et al., 2023) Hemlata and et.al, prepared silver nanoparticles by the aqueous leaf extract of C. prophetarum. (Meena et al., 2020) Shahzad and co-workers have prepared the AgO nanoparticles by Biological, Chemical and Physical Methods. (Mughal & Hassan, 2022) By aqueous extracts of fresh leaves of Jasminum officinale, Elhawary and co-workers...
synthesized the Ag NPs, this process was used as reduction factor to produce ecofriendly silver nanoparticles, and this method a safe and consequently does not have side effects. ([Elhawary et al., 2022])

In this study described the preparation of AgO NPs by two approaches, then the properties of the products were compared to each other by many techniques.

**Experimental part**

**Biological method**

*Preparation of plant leaves extract and synthesis of silver oxide nanoparticles:* The leaves castor plant was obtained from trees at Diyala city, they were washed well with tap water followed by deionized water then air dried. The leaves were cut and ground well, and stored in dry conditions. 5 g of powder castor leaves were added to deionized water (75 mL) and boiled at 80 °C for 3 hours until the colour of solution changed and then the mixture was cooled to room temperature and filtered using whatman paper (No.1) to obtain a clear filtrate. Centrifugation the filtrate at (1200) rpm for (45) minutes to remove all fine plant biomaterials and used it in the preparation of the AgO nanoparticles. The salt solution was synthesis via dissolving a 3.0 g (AgCl\(_2\)) in 150 mL of deionized water. The extract solution was gradually added to the salt solution with stirring at (40 °C), the temperature of the solution was raised to (80 °C) under stirring and sodium hydroxide solution (0.1 M) was added dropwise to the mixture (brine and leaves extract), to obtain the number of pH at approximately 12, the mixture was left at constant stirring for 2 hours. Then, the mixture was filtered; the precipitate was collected and washed with deionized water and ethanol to reach a pH of approximately 7 and obtain silver oxide nanoparticles AgO NPs, as shown in figure 1.

**Chemical method**

AgO nanoparticles were synthesized via co-precipitation method using silver chloride (AgCl). In 100 mL deionized water was dissolved AgCl\(_2\) salt (2 g, 0.013 mol), then, sodium hydroxide solution (0.1 M) was slowly dropped with stirring to obtained PH at 12. White precipitate from AgOH was obtained, and several times was washed by deionized water and absolute ethanol until pH reached 7, subsequently, dried it at 80 °C for 5 h. Finally, calcination of AgOH in a furnace at (500°C) for (5) hours to obtained silver oxide nanoparticles (AgO NPs).

**RESULT AND DISCUSSION**

The first evidence of biosynthesis synthesis of Ago NPs is the colour change of the silver chloride salt solution during successive time periods 5, 15 and 25 min and 24 hours, dark colour can be seen at regular intervals, as shown in figure 2. ([Ayad et al., 2019])
Characterization of synthesized silver oxide nanoparticles: Synthesis rout with NaOH solution as precipitation agent was taken to study the synthesis of silver oxide nanoparticles by two methods, and were analyzed using UV spectroscopy, X-ray diffraction, scanning and transmission microscope of resonance band at around 300–450 nm.

**UV-Vis Analysis of AgO NPs**

The Haiss equation was used to estimate the size of AgO nanoparticles, by help the surface Plasmon resonance properties, which has exploited in optical spectroscopy. (Jawad et al., 2019)

$$d = \frac{\ln (\lambda_{SPR} - \lambda^0)}{L_1 - L_2}$$

Where: $d$ is the particle diameters of the AgO NPs, $\lambda_{SPR}$ is wavelength at which maximum absorption occurs, $\lambda^0$ is wavelength at which minimum absorption occurs, $L_1$ and $L_2$ are parameters equal 6.53 and 0.0216, respectively. Repeating the centrifugation at 10000 rpm for 30 min the obtained nanoparticle solution was purified, and then re-dispersion of the AgO NPs in deionized water in the ultrasonic at 45 min. Figure 3 show the optical absorption spectra of prepared AgO NPs solutions using chemical and eco-friendly method. The UV-Vis spectra reveal characteristic absorption peaks of the AgO NPs shown in table 1. From table 1 the effect of the method type on the size of silver oxide nanoparticles clearly, while the silver nanoparticles sizes unaffected which were similar.

**XRD analysis of AgO NPs**

The size of AgO nanoparticles have calculated from Scherrer formula as:

$$D = \frac{K\lambda}{\beta \cos \theta}$$
Where D is Average Crystalline size, \( \lambda \) is the wavelength of the x-ray source, \( \beta \) is the full-width at half-maximum (FWHM) in radian and \( \theta \) is the Bragg’s angle. The XRD pattern of AgO NPs was shown in Figure (4). The XRD factors as shown in tables (2). The AgO and Ag phase was a dominated, with cubic structure. It can be noticed that the peak are broad, indicating the formation of nanocrystalline silver and silver oxide phase. Many factors of XRD such as dislocations and micro strain (\( \varepsilon \)).

The dislocation (\( \delta \)) and micro strain (\( \varepsilon \)) parameters have calculated using; (Fall et al., 2021)
\[
\delta = \frac{1}{D^2} \\
\varepsilon = \beta \cdot 4 \tan \theta
\]
The dislocation density is a measure of the number of dislocations in a unit volume of a crystalline material. micro strain (\( \varepsilon \)) is a force or influence that stretches, pulls or put pressure on something, sometimes causing damage.

The number of crystallites (N), which determined using the following formula,
\[
N = \frac{1}{D^3}
\]
The silver oxide nanoparticles AgO NPs was characterized using powder XRD. In which to confirm the particles of silver oxide and to know the structural information for product. Figure 4 show the XRD pattern of silver oxide nanoparticles that synthesized by chemical and eco-friendly method. The pattern of AgO NPs for chemical method clearly shows the main peaks at \( (2\theta) \) 38.26°, 44.47°, 64.71° and 77.73° corresponding to the (111), (200), (220) and (311) planes, respectively. The typical pattern of chemical synthesized AgO NPs was found to possess a cubic structure with average crystalline size 23.04 nm.

![Fig.4 XRD pattern of synthesized AgO NPs by chemical and eco-friendly method.](image)

While, the data of synthesized AgO NPs using castor leaves extract is shown in figure 4, the main peaks at \( (2\theta) \) 28.06°, 32.47°, 38.38°, 44.52°, 46.39°, 55.10°, 57.71°, 64.74° and 77.61° corresponding to the (111), (200), (220) and (311) planes, respectively. Additionally, the AgO NPs have found to possess a cubic structure with crystalline average size 17.57 nm. In addition, three unassigned peaks appeared at \( (2\theta) \) 38.26°, 44.47° and 77.73° for AgO NPs, these peaks were weaker than those of silver oxide nanoparticles.

**Table (2)** show parameters obtained from XRD pattern of the AgO NPs.

<table>
<thead>
<tr>
<th>Method</th>
<th>D, nm</th>
<th>( \delta ) ( \times 10^8 ), nm(^2)</th>
<th>( a ), Å ( \times 10^3 ) m(^3)</th>
<th>( c ) ( \times 10^3 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>chemical</td>
<td>23.04</td>
<td>2.09</td>
<td>4.23</td>
<td>0.314</td>
</tr>
<tr>
<td>Eco-friendly</td>
<td>17.57</td>
<td>1.83</td>
<td>4.11</td>
<td>0.527</td>
</tr>
</tbody>
</table>

The illustrated figures showed that there was a clear effect of the of method type that used, which had a role in the affecting the average size of the silver oxide nanoparticles. Accordingly, the comparing of X-ray diffraction pattern of prepared silver oxide nanoparticles using castor extracts and chemical method showed clearly effect on the average diameter size of silver nanoparticles. See table 1.

28
FE-SEM images of AgO NPs
FE-SEM technique has used to investigate the morphology of the AgO NPs. Figures 5 and 6 with different magnifications give FE-SEM images of AgO NPs, which synthesized by chemical and Eco-friendly method, respectively. Figure 5 illustrate the FE-SEM images of synthesized AgO NPs using chemical methods confirmed the presence of irregular shapes and agglomeration nanoparticles with average size 122 nm. While, FE-SEM image for the synthesized AgO NPs using castor extracts shown in the figure 6. FE-SEM images of the synthesized AgO NPs were confirmed the presence of spherical shapes of AgO nanoparticles with average size 57 nm.

![Fig. 5 FE-SEM images of AgO NPs synthesized chemical method.](image1)

![Fig. 6 FE-SEM images of AgO NPs synthesized Eco-friendly method.](image2)

TEM images of AgO NPs
For measurement of the size and shape of AgO nanoparticles use TEM, which is gave advanced analytical for imaging. TEM Image of AgO NPs synthesized by the chemical and eco-friendly method was shown in Figures 7 and 8, respectively. The spherical shape and irregular aggregation, the dominant characteristic of AgO nanoparticles with average nanoparticles size about [50 – 200] nm. ([Fall et al., 2021], (Ali et al., 2023)
CONCLUSION
In this work, the synthesis of stable silver oxide nanoparticles using aqueous castor leaves extract as reducing and stabilizing agent. Pure Silver oxide nanoparticles (AgO NPs) were synthesized successfully using chemical method. The spherical and irregular shape synthesis of AgO NPs has confirmed by XRD analysis, FE-SEM and TEM with different average crystalline size. UV spectra confirmed that AgO NPs have different diameter size. That confirmed the type of method used had a clear effect on the shape and size of silver oxide nanoparticles.

REFERENCES


