



Chemical Precipitation-assisted Synthesis of Ag₂O Nanoparticles: Structural, Optical and Morphological Insights

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ABSTRACT

Initially created through an uncomplicated chemical precipitation approach, Silver Oxide Nanoparticles (Ag₂O NPs) were thermally treated over a wide range of temperatures (150°C - 600°C) to explore how the varying thermal changes affected the morphology and crystal structure properties of these nanoparticles. Ultraviolet visible spectroscopic characterisation proved that nanoparticles were made with a distinctive surface plasmon resonance peak at 432nm. The analysis of X-ray diffraction data revealed that higher annealing temperatures were related to the formation of a monoclinic Ag₂O phase with improved crystallinity. The increase in crystallite size from 10.25nm to 30.11nm indicated that there are temperature-controlled thermally-induced grain growth processes at these elevated temperatures were affecting crystallite sizes in a highly significant manner, as a result of these thermally-induced processes. Scanning Electron Microscopy proved that particles were most spherical in shape, widely-spaced and had a minimal degree of aggregation. Fourier Transform Infrared Spectroscopy showed the presence of Ag-O vibrational bands confirming that Oxides were produced. The results of this study showed that the annealing/thermal processing of Ag₂O NPs is a critical parameter to adjust crystallinity and particle size which demonstrates the techniques suitability for scalable synthesis of Ag₂O NPs for potential biological and catalytic applications.

Keywords: Silver; Nanoparticles; crystallization; Synthesis; Oxides.

1. INTRODUCTION

Nanoparticles are receiving attention in many industries because they are useful in various applications, including biology, catalysis, optics, pharmaceuticals, health care, agriculture, and many industrial processes (Abdelmigid *et al.* 2022). Commonly used methods for creating nanoparticles include physical and chemical methods; unfortunately, these methods have significant drawbacks, such as high costs to operate and produce nanoparticles, require high temperatures/pressures, and utilize toxic materials that may be hazardous to the environment and human health (Jamkhande *et al.* 2019). Therefore, new environmentally friendly and sustainable methods of producing nanoparticles have garnered increased attention (Alhujaily *et al.* 2022). One area of particular interest is using plants or biogenic materials to produce nanoparticles (e.g., silver nanoparticles [AgNPs]) quickly, simply, and effectively without using harmful chemicals, as in traditional methods (Wang *et al.* 2016).

Many different organisms can be used to create AgNPs, including macroalgae and microalgae, and AgNPs have been shown to exhibit interesting properties

such as antibacterial, antioxidant, cytotoxic and anticancer activity (Dash *et al.* 2012). The increased interest in AgNPs reflects their unique properties due to silver's chemical stability and the versatility of silver for use in therapeutic and biochemical applications. In most biological systems, the synthesis of AgNPs is accomplished using biomolecules, such as enzymes and metabolites, that facilitate the reduction of silver ions to form nanoscale particles (Dhavale *et al.* 2020). Silver oxide is known for its ability to act as a sensor for various chemicals, such as ammonia and carbon monoxide. It is also known to catalyze reactions involving the oxidation of ethylene and methanol (Bielmann *et al.* 2002). Silver oxide nanoparticles exhibit good fluorescence (Jiang *et al.* 2005) and optical properties (Vaseashta and Dimova-Malinovska, 2005) and have the potential to be used in data storage devices of the future. Their property of optical fluorescence memory facilitates their usage in optical media to enhance the readout signal by efficient utilisation of laser energy. Silver oxide nanostructures also have the potential to be used in photovoltaic devices, (Zaidi *et al.* 2025) optical storage devices (Her *et al.* 2004) and plasmon photonic devices (Tominaga, 2003). They can also be used as active cathode materials for zinc/silver oxide batteries (Dallek *et al.* 1986). Until now,

silver oxide nanoparticles have been prepared using reactive sputtering (Buchel *et al.* 2001) and electrochemical/thermochemical (Wei *et al.* 2011) means. These have resulted in development of techniques for relatively inexpensive silver oxide nanostructures, but require specialised equipment and involve a variety of chemicals that could have an impact on the environment.

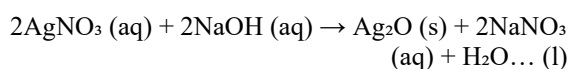
2. EXPERIMENTAL DETAILS

2.1 Materials

Sodium hydroxide (NaOH) pellets were acquired from Lobachemie (India), while silver nitrate Silver Nitrate (Merck - India) was utilized in this study and all reagents were analytical grade and were not additionally purified. Cleaning and making solutions during experimentations used distilled and deionized water.

2.2 Synthesis

The Silver Oxide Nanoparticles (Ag₂O NPs) were synthesised by the method of chemical precipitation per the standard procedure (Mir *et al.* 2026). First dissolving 5 g of silver nitrate and 5.4 g of sodium hydroxide in small amounts of water, these two separate solutions were poured into a beaker and then stirred at room temperature to ensure total dissolution. Next, as the sodium hydroxide solution was slowly added dropwise into the silver nitrate solution, the mixture was continuously stirred. The formation of silver oxide nanoparticles was characterized by a slow colour change of the reaction, starting as dark brown ending with a brownish black colour and then finally black. Upon collection of the resultant black precipitate by centrifugation using Eppendorf refrigerated centrifuge instrument (Model 5702R - Germany), any leftover sodium nitrate was washed away from the precipitate using ethanol followed by deionised water repeatedly. The purified sample was dried in an oven (approximately 50°C) to enhance crystal growth and then subjected to a post-drying annealing step at either 1500°C, 300°C, 450°C, or 600°C. Following this, the annealed product was finely ground to be used for further characterization. The reaction can be summarized as follows:



2.3 Characterization of Silver Oxide Nanoparticles

2.3.1 UV-VIS Spectroscopy

The colour changes of the reaction mixture indicated that Ag₂O dispersions had formed. The UV-Visible spectra were acquired using a Shimadzu UV-2500 spectrophotometer (Japan) at regular time intervals

(every 3 mL) over the range of 200–800 nm of wavelength (nm) with a resolution of 1 nm.

2.3.2 Fourier Transform Infra-Red (FT-IR) Spectroscopy Analysis

FT-IR spectroscopy was employed to identify functional groups that were present in the formation of the nanoparticles. To acquire the FT-IR spectra a combination of 20 mg of FT-IR grade KBr and 2 mg of the dried nanoparticle sample were mixed and ground to create a small pellet for measurement. The FT-IR spectra were recorded using an AVATAR 300 FT-IR spectrometer (Thermo Nicolet USA) over the range of 4500 – 450 cm⁻¹.

2.3.3 X-Ray Diffraction (XRD) Analysis

To identify the crystalline structure and quantify the crystallite size of the synthesized nanoparticles, XRD analysis was performed using a Bruker D8 Advance diffractometer (Germany) and Cu K α radiation (λ = 0.15405 nm). The XRD patterns were obtained in the range of 2 θ from 10 ° to 100 degrees and at 40 kV (kilovolts, voltage) and at 40 mA (milliamperes, current). The scan rate was 0.25 min⁻¹ (minutes, per minute) and the step size used was 0.015 degrees.

2.3.4 Energy Dispersive X-Ray and Scanning Electron Microscopy (SEM-EDX) Imaging

The morphology and elemental composition of the nanoparticles were obtained using SEM and EDX imaging. A JEOL JSM-7600F microscope (Japan) equipped with an EDX detector was used to characterize nanoparticles that were deposited as drops of nanoparticle suspension on aluminum-coated grids and allowed to dry.

3. RESULTS AND DISCUSSION

3.1 Visible UV Spectroscopy

The presence of an evident broad absorption band in the VIS region of the UV-VIS spectrum confirms the formation of the AgO (silver oxide) NPs as well as the specific features of the silver oxide (AgO) NPs through their characteristic absorption maximum (λ_{max}) at 432 nm approx. as a result of surface plasmon resonance (SPR). The resonance (SPR) band indicates the particle's dimensions and optical characteristics associated with the collective oscillation of conduction band electrons due to incident light on the silver oxide NPs (Ali *et al.* 2011). The UV-VIS spectral absorption exhibits a consistent increase in absorbance from the UV range of 300–350 nm to 432 nm for the SPR absorption maximum and decreasing absorbances in the higher wavelength regions from approx. 500–800 nm, suggesting very little to no aggregation and relatively uniform particle distribution. The width of the SPR peak

indicates the size distribution of nanoparticle sample and the existence of potential polydispersity.

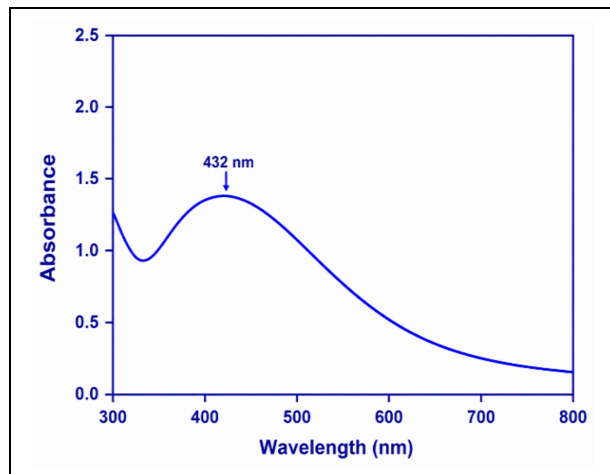


Fig. 1: The UV-Visible spectra of Ag₂O-NPs

3.2 Analysis of XRD

X-ray Diffraction (XRD) patterns (Figure 2) reveal the crystalline structure of Ag₂O nanoparticles after they were treated with heat at 150°C, 300°C, 450°C, and 600°C. The Ag₂(OH)₂ phase formed when dried at 50°C and is also represented in the XRD (Figure 2). To validate the development of the crystalline structure, the diffraction peaks of the material following heat treatment were compared with the corresponding diffraction patterns from accredited reference sources (International Centre for Diffraction Data, 2000) as shown in table 1. Figure 3 illustrates the XRD pattern of the disilver oxide (Ag₂O) sample (-60°C) and confirms that the crystalline Ag₂O phase formed as identified in the previous figure. The high degree of crystallinity for this phase can be confirmed due to the near-matching diffraction peak positions and relative intensities with the currently available data for monoclinic Ag₂O (Radhakrishnan and Beena, 2014). The Ag₂O samples treated with heat at either 150°C or 350°C also demonstrated similar crystallinity characteristics as the established patterns. In contrast, the samples treated with heat at either 450 or 600 °C reflected the greatest intensity peaks in addition to demonstrating improvements in crystallinity due to the more sharp and well-defined nature of the diffraction peaks as the thermal processing temperature increased. In addition, while increased order within the atomic arrays can be attributed to higher heat treatment temperatures, there was partial crystallization at the lower temperature of 150°C. The degree of crystallization was determined primarily by heating the samples to 600°C and having the final result as an almost completely developed phase.

An overview of how the sample was thermally treated is characterised by an analysis of the FWHM (β) measurements (Table 2). The crystal structure of the 600°C samples supports a base-centred monoclinic unit

cell by its computed crystal cell sizes (a, b and c) and the measured FWHM values found in the reference Ag₂O (International Centre for Diffraction Data, 2000). With increased annealing temperatures we see an increase in average crystallite size (Table 3). This increase can be attributed to increased atomic diffusion which occurs at high temperatures and thereby promotes the coalescence and continued growth of the coalesced particles. This will happen as thermal energy continues to increase with the addition of more thermal energy to the original particle size, leading to an increase in average crystallite size. Therefore, higher levels of annealing will favourably affect the degree of oxidation and overall development of the crystallites present in Ag₂O nanoparticles (Raksa *et al.* 2005).

Table 1. shows the peak intensities and relative peak intensities of the most noticeable peaks in the Ag₂O-NPs XRD patterns that were annealed at 1500C, 3000C, 4500C, and 6000C

Sample Condition	Diffraction Intensity (Counts)	Normalized Intensity (%)
Ag ₂ O NPs heat-treated at 150°C	1010	23.4
Ag ₂ O NPs heat-treated at 300°C	1853	31.4
Ag ₂ O NPs heat-treated at 450°C	2811	47.3
Ag ₂ O NPs heat-treated at 600°C	5121	99.1

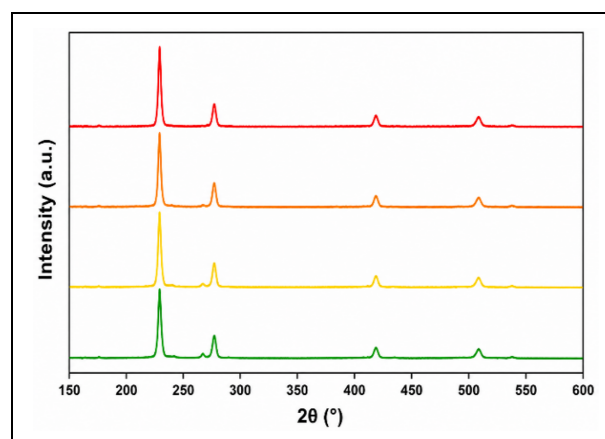


Fig. 2: XRD patterns of Ag₂O-NPs and Ag₂(OH)₂ complex annealed at 1500C, 3000C, 4500C, and 6000C

3.3 Evaluation of Scanning Electron Microscopy

SEM Images of an Ag₂O Nanoscale Structure that was Thermally Treated (Annal Logarithm Scale) as shown in figure 4. The Ag₂O NPs appear to have a mostly spherical shape and a relatively uniform distribution in terms of the image that was captured at a SEM magnification of 27,000 times on the surfaces of the Ag₂O NPs. In general, metal oxides will exhibit a highly

homogeneous and well-defined crystal shape; however, there will also be some evidence of particle agglomeration in the SEM image. Additionally, the SEM image shows that multiple small spherical shaped groups of particles are enough together; thus, some of the groups will contain several of the same spherical shaped particles, forming a group of regularly shaped polyhedrons. This image also suggests a growth mode of an island-like type from the highly regular spherical shaped NPs. The images of the particles indicate a process of particle coalescence has occurred, which is shown by the presence of larger particles that appear to be surrounded by smaller particles as a result of the thermal treatment applied to the nanoparticles used in this study. The SEM images of the Ag₂O NP sample presented in this study are consistent with previous studies on the morphology of Ag₂O NPs (Radhakrishnan and Beena, 2014).

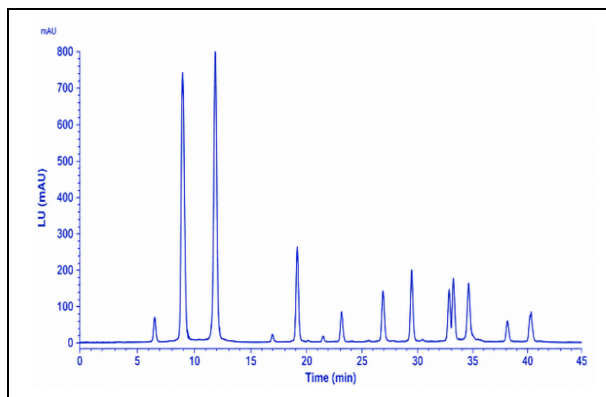


Fig. 3: Ag₂O-NPs annealed at 600°C show their XRD patterns the location and relative intensity of the 05-0661 JCPDS card file diffraction peaks for the monoclinic phase are shown by the vertical lines (l)

Table 2. shows the lattice parameters (a, b, and c) and complete width at half the maximum intensity, β , for Ag₂O-NPs that were annealed at 1500C, 3000C, 4500C, 6000C and standard

Sample Description	Lattice Parameter a (nm)	Lattice Parameter b (nm)	Lattice Parameter c (nm)	Monoclinic Angle β (°)
Ag ₂ O nanoparticles heated at 150°C	0.463439	0.341231	0.502134	98.9215
Ag ₂ O nanoparticles heated at 300°C	0.465489	0.341311	0.502144	98.9385
Ag ₂ O nanoparticles heated at 450°C	0.466211	0.341430	0.501304	98.9590
Ag ₂ O nanoparticles heated at 600°C	0.467213	0.341124	0.501695	98.9715
Reference (Ag ₂ O)	0.468210	0.341290	0.502140	98.9799

Table 3. shows the average crystallite size of Ag₂O-NPs that were annealed at 150, 300, 450 and 6000Celsius

Sample Description	Mean Particle Size (nm)
Ag ₂ O nanoparticles heated at 150°C	11.32
Ag ₂ O nanoparticles heated at 300°C	17.32
Ag ₂ O nanoparticles heated at 450°C	19.14
Ag ₂ O nanoparticles heated at 600°C	29.10

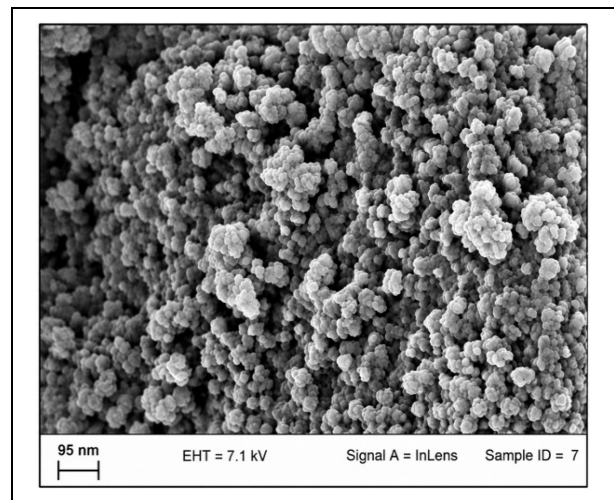


Fig. 4: Ag₂O-NPs annealed at 600°C as seen in a SEM image

3.4 Energy Dispersive X-ray (EDX)

Figure 5 shows that EDX has identified two major elements present in Ag₂O nanoparticles (Ag₂O) after annealing at 600 °C; these include O and Ag. EDX was performed at an acceleration voltage of 10 keV. Analysis provides strong evidence that silver oxide nanoparticles have successfully formed since O constitutes approximately 35 wt% and Ag approximately 65 wt%. Based on the lack of additional peaks found on the EDX spectrum, it also supports there are no other contaminants present in the sample analyzed and indicates near-stoichiometric ratios within the elemental composition supporting the synthesis of pure Ag₂O. The above-mentioned EDX profiles are supported by corresponding XRD results and coincide with previously published reports (Vaseashta and Dimova-Malinovska, 2005).

3.5 Examination of FTIR Analysis

As illustrated in Figure 6 (FTIR) for Ag₂O NPs that were heat treated at 600°C, the broad absorption band observed at approximately 3454 cm⁻¹ is due to the stretching vibrations of -OH groups which arise from adsorbed water on the surface of the nanoparticles. This is typical for nanostructured materials due to the

relatively large surface to volume ratio. The absorption peak at 2919 cm^{-1} is related to C–H (carbon-hydrogen) stretching vibrations of residual organic matter on the surface of the NPs. The region between 2690 to 3800 cm^{-1} is attributed to O–H stretching modes. The band at approximately 1635 cm^{-1} could be due to Ag–O interactions or to the bending modes of surface species associated with the NPs. Two distinct peaks are present in the lower wavenumber band (500 – 700 cm^{-1}) near 540 cm^{-1} and 575 cm^{-1} ; these bands confirm the presence of silver oxide and represent Ag–O stretching vibrations. The band near 540 cm^{-1} corresponds to the unique vibrational mode (B_{2u}) and could be attributed to variations in NP size and surface effects that cause small displacements in the peak position. The unique vibrational characteristics of metal-oxygen bonds contribute to the formation of Ag_2O nanoparticles in a monoclinic structure. No additional bands are observed in this spectral region, indicating that no silver oxide phases other than Ag_2O exist. The successful formation of Ag_2O nanoparticles is supported by the FTIR spectra, which generally agree with the results from the literature.

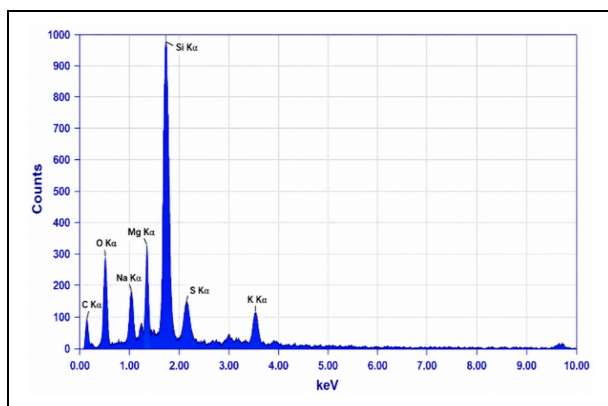


Fig. 5: Ag_2O -NPs annealed at 600°C were analyzed by energy dispersive X-ray

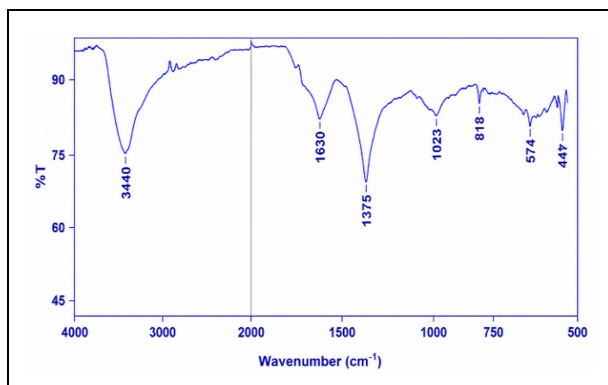


Fig. 6: Ag_2O -NPs annealed at 600°C and their FTIR spectra

4. CONCLUSION

A simple precipitation strategy for generating silver oxide (Ag_2O) nanoparticles was utilized and

followed by the systematic modification of such nanoparticles using the technique of thermal annealing. Increased cremation temperatures lead to increased levels of atomic diffusion and therefore increased crystalline structure of particles; thus, providing better quality particles via the growing of grains. The formation of a single crystalline phase Ag_2O nanocrystal (monoclinic structure) was verified using X-ray diffraction (XRD), and furthermore the presence of several high-quality nanoparticles and two distinct morphologies were confirmed using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) respectively. The experimental results emphasize the importance of thermal treatment as an effective method for modifying properties of nanoparticles. This method can be used to produce Ag_2O nanoparticles with tunable properties on a large scale and therefore has potential applications in biomedical, sensing, and catalytic areas.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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