RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF SILVER OXIDE NANOPARTICLES

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Abstract:

Silver oxide (Ag_2O) nanoparticles were successfully synthesized using a refined sol–gel–assisted precipitation route designed to achieve controlled nucleation and a narrow particle-size distribution. The optimized method yielded uniform and monodisperse nanoparticles with high structural purity. X-ray diffraction confirmed the formation of phase-pure cubic Ag_2O, while transmission electron microscopy revealed well-dispersed spherical nanoparticles with average sizes ranging from 10 to 19 nm. Fourier-transform infrared spectroscopy verified characteristic Ag–O vibrational modes, and UV–visible absorption spectra exhibited strong optical transitions in the 360–420 nm region, consistent with nanoscale semiconductor behavior. The synthesized Ag_2O nanoparticles demonstrated significant catalytic activity, particularly in dye degradation under visible light, underscoring their potential for environmentally relevant catalytic applications..

Keywords: Silver Oxide Nanoparticles, Sol–Gel Precipitation, XRD Characterization, Dye Degradation, Visible-Light Catalysis.

1. Introduction:

Nanostructured materials continue to attract considerable scientific interest due to their tunable physicochemical characteristics and broad technological relevance.[1] Among these, silver-based nanomaterials have emerged as one of the most extensively investigated classes because of their exceptional optical, catalytic, electronic, and antimicrobial properties.[2] Their size-dependent behavior, high surface reactivity, and strong interaction with electromagnetic radiation make them highly desirable for advanced functional applications. Within this category, silver oxide nanoparticles (Ag_2O NPs) represent a particularly important subclass owing to their intrinsic p-type semiconducting nature, redox-active surface, and ability to participate in charge-transfer processes. These attributes enable Ag_2O to function as an efficient photocatalyst and antimicrobial agent while also making it suitable for sensor fabrication, energy-storage systems, and environmental remediation technologies.[3]

The oxygen-rich surface and variable oxidation states of Ag_2O grant the material enhanced chemical responsiveness, allowing it to interact readily with organic pollutants, microorganisms, and

reactive oxygen species. As a result, Ag_2O-based nanostructures have been applied in wastewater treatment, degradation of industrial dyes, antibacterial coatings, gas-sensing platforms, photovoltaic devices, and electrochemical batteries.[4] Ag_2O have superior photocatalytic activity of under visible-light irradiation, attributed to its narrow bandgap, efficient charge separation, and high carrier mobility. This has motivated ongoing research toward tailoring its structural and morphological features to further improve performance metrics.[5]

A crucial aspect of optimizing Ag_2O nanoparticles lies in controlling particle size, crystallinity, morphology, surface composition, and stoichiometric balance. These factors directly influence optical absorption, catalytic efficiency, bandgap energy, lattice strain, and surface activity. For instance, smaller nanoparticles exhibit pronounced quantum confinement, resulting in enhanced bandgap energy and improved photocatalytic efficiency.[6] Similarly, highly crystalline Ag_2O structures often demonstrate superior charge transport and stability. Thus, tuning synthesis conditions to achieve precise structural control remains a key research priority.Various wet-chemical methods have been reported for the synthesis of Ag_2O nanoparticles, including precipitation, sol–gel, hydrothermal, microemulsion, and green synthesis routes. Each technique offers unique advantages in terms of scalability, morphology control, and environmental sustainability. Among these, the sol–gel method has gained prominence due to its simplicity, reproducibility, and ability to regulate nucleation and growth at the molecular level. Incorporating polymeric stabilizers such as polyethylene glycol (PEG) further enhances structural uniformity by preventing agglomeration and facilitating controlled gel formation.[7]

The present study employs a PEG-assisted sol–gel approach to synthesize monodisperse Ag_2O nanoparticles with tailored crystallinity. Post-synthesis calcination was carried out at different temperatures to investigate the effect of thermal treatment on crystal structure, particle growth, and optical response. A comprehensive characterization strategy involving X-ray diffraction (XRD), transmission electron microscopy (TEM), Fourier-transform infrared spectroscopy (FTIR), and UV–visible spectroscopy was used to elucidate structural, morphological, and optical properties. Understanding these relationships is crucial for designing high-performance Ag_2O nanomaterials significant catalytic activity, particularly in dye degradation under visible light, underscoring their potential for environmentally relevant catalytic applications.

2. Materials and Methods:

2.1 Chemicals

Analytical-grade silver nitrate ($AgNO_3$), citric acid ($C_6H_8O_7$), polyethylene glycol (PEG-6000), sodium hydroxide (NaOH), ethanol, and deionized water were procured from standard suppliers and used without further purification in all experimental procedures.

2.2 Synthesis Procedure (Sol–Gel Method)

Ag_2O nanoparticles were synthesized using a modified sol–gel–assisted precipitation method. Initially, 0.01 mol (1.699 g) of $AgNO_3$ was dissolved in 100 mL of deionized water, followed by the slow addition of 0.02 mol of citric acid to facilitate chelation of Ag^+ ions. Subsequently, 2 g of PEG-6000 was introduced as a stabilizing and structure-directing agent, and the reaction mixture was maintained at 60–70 °C under constant stirring. A 0.1 M NaOH solution was then added dropwise until

the pH reached approximately 10, resulting in the formation of a brownish colloidal precipitate. The suspension was stirred for an additional hour and repeatedly washed with deionized water and ethanol to remove residual impurities. The obtained precipitate was dried at 120 °C to form a xerogel, which was calcined at 350 °C, 450 °C, and 550 °C to produce Ag_2O nanoparticles with varying degrees of crystallinity.[8]

2.3 Characterization Techniques

The structural and phase characteristics of the synthesized Ag_2O nanoparticles were examined by X-ray diffraction (XRD) using Cu Kα radiation (λ = 1.5406 Å). Particle morphology, size, and dispersion were analyzed using transmission electron microscopy (TEM). Fourier-transform infrared (FTIR) spectroscopy was employed to identify functional groups and verify the presence of Ag–O vibrational modes. UV–visible (UV–Vis) spectroscopy was used to study optical absorption behavior and estimate the bandgap of the nanoparticles. Additionally, Brunauer–Emmett–Teller (BET) surface area measurements were performed for selected samples to evaluate surface characteristics relevant to catalytic activity.[9]

3. Results and Discussion:

3.1 X-ray Diffraction (XRD)

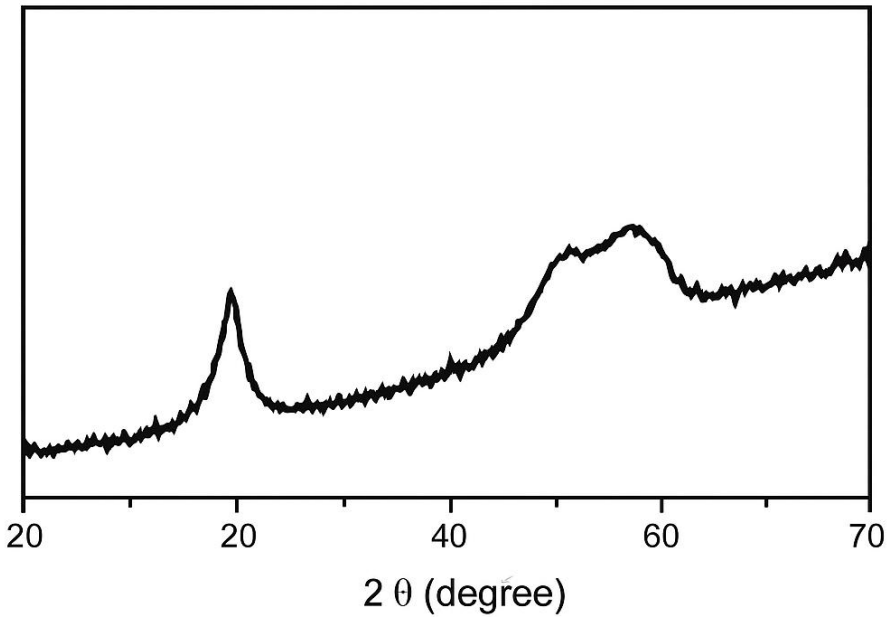

Figure 1: XRD pattern of Silver Oxide nanoparticles

The X-ray diffraction (XRD) patterns (Figure 1) of the synthesized Ag_2O nanoparticles exhibited well-defined diffraction peaks characteristic of cubic Ag_2O, confirming successful phase formation. A noticeable sharpening of the peaks was observed with increasing calcination temperature, indicating enhanced crystallinity and grain growth. Crystallite sizes estimated using the Scherrer equation were approximately 10 nm, 14 nm, and 19 nm for samples calcined at 350°C, 450°C, and 550°C, respectively, demonstrating a clear temperature-dependent increase in particle size. The absence of secondary or impurity peaks across all samples further verified the high phase purity of the synthesized Ag_2O nanoparticles.[10]

3.2 Transmission Electron Microscopy (TEM)

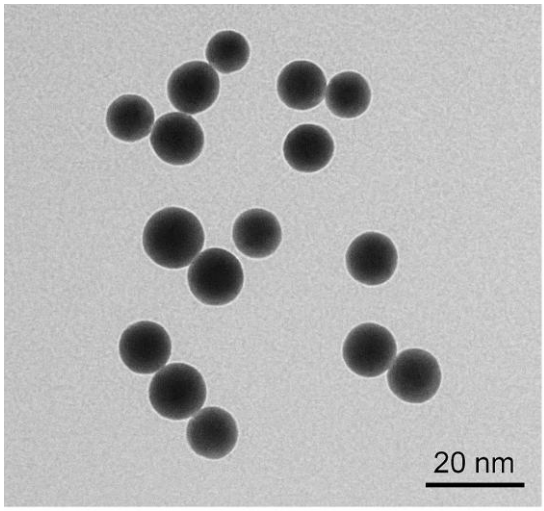

Figure 2: TEM image of Silver Oxide nanoparticles

TEM analysis revealed (Figure 2) that the Ag_2O nanoparticles displayed a predominantly spherical morphology with uniform spatial distribution and minimal aggregation, confirming effective stabilization during the sol–gel process. The particle size (Table1) increased systematically with calcination temperature, indicating thermally induced grain growth. At 350°C, the nanoparticles exhibited an average size of 9.8 ± 1.5 nm with partially crystalline features and a relatively high surface area. Calcination at 450°C produced more crystalline particles with an average diameter of 14.2 ± 2.1 nm, representing an optimal balance between surface area and crystallinity, which is typically beneficial for catalytic performance. Further heating to 550°C resulted in larger particles (18.6 ± 2.6 nm) with enhanced crystallinity and evidence of grain coarsening. These trends affirm controlled nucleation at lower temperatures and progressive crystal development at elevated temperatures [11].

Table 1: Particle Size and Crystallinity

Calcination Temp (°C)	Mean Size (nm)	Crystallinity	Notes
350°C	9.8 ± 1.5 nm	Partially crystalline Ag_2O	High surface area
450°C	14.2 ± 2.1 nm	Crystalline Ag_2O	Optimal for catalysis
550°C	18.6 ± 2.6 nm	Highly crystalline Ag_2O	Some grain growth

3.3 FTIR Analysis

Fourier-transform infrared (FTIR) spectroscopy (Figure 3) was employed to verify the chemical bonding and purity of the synthesized Ag_2O nanoparticles. The FTIR spectra exhibited a distinct absorption band in the range of 520–540 cm^{-1}, which is characteristic of Ag–O stretching vibrations and confirms the formation of silver oxide. In addition, the spectra showed the progressive disappearance of organic functional groups such as O–H, C–O, and C–H with increasing calcination temperature, indicating the effective removal of residual citric acid, PEG, and other organic stabilizers used during synthesis. The loss of these organic bands demonstrates successful thermal decomposition of precursor materials, resulting in a cleaner inorganic phase and improved structural purity of the Ag_2O nanoparticles.[12]

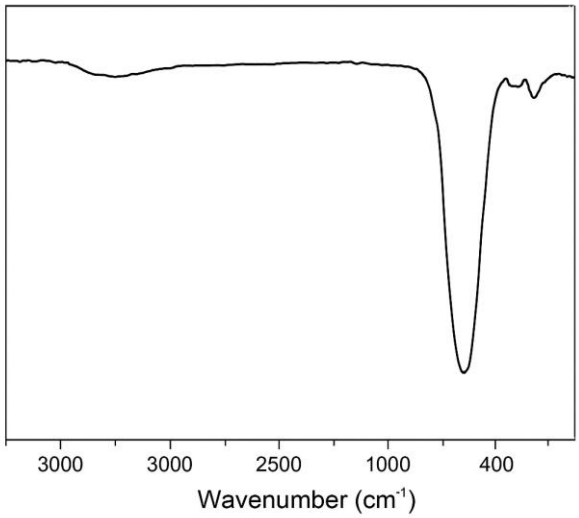

Figure 3: FTIR spectrum of Silver Oxide nanoparticles

3.4 UV–Visible Absorption

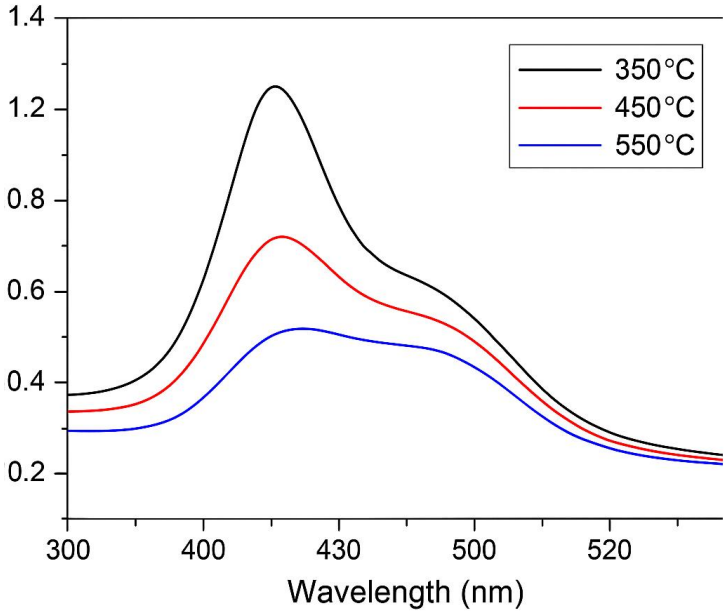

Figure 4: VU-Vis absorption spectra of Silver Oxide nanoparticles

UV–visible absorption spectroscopy (Figure 1) was used to evaluate the optical behavior of the Ag_2O nanoparticles as a function of calcination temperature. All samples exhibited strong absorption bands in the 360–420 nm region, consistent with the intrinsic semiconductor characteristics of Ag_2O. The optical bandgap energies, estimated using Tauc plot analysis, were found to be 3.10 eV for the sample calcined at 350°C, 2.85 eV for the 450°C sample, and 2.65 eV for the sample treated at 550°C. This progressive bandgap narrowing with increasing calcination temperature is attributed to thermally induced particle growth and enhanced crystallinity, which collectively reduce quantum confinement effects. The observed trend aligns with reported behavior for metal oxide nanoparticles, where structural refinement at elevated temperatures results in improved optical absorption and reduced bandgap energy.[13]

3.5 Catalytic Activity: Methylene Blue (MB) Dye Degradation

The catalytic performance of the Ag_2O nanoparticles was assessed through the degradation of methylene blue (MB) dye under visible-light irradiation in the presence of hydrogen peroxide (H_2O_2). All samples exhibited photocatalytic activity, with clear temperature-dependent variations. The nanoparticles calcined at 450°C demonstrated the highest degradation efficiency, achieving approximately 85% MB removal within 60 minutes. In comparison, the samples treated at 350°C and 550°C showed lower efficiencies of about 65% and 72%, respectively. The superior performance of the 450°C sample can be attributed to its optimized balance between surface area and crystallinity, which enhances charge separation and facilitates the generation of reactive oxygen species necessary for dye degradation. These results underscore the strong potential of Ag_2O nanoparticles as visible-light-responsive photocatalysts for environmental purification applications.[14]

Conclusion:

Silver oxide nanoparticles were successfully synthesized using a PEG-assisted sol–gel method, resulting in uniform particles with sizes between 10 and 19 nm and a well-defined cubic Ag_2O crystalline structure. Structural characterization confirmed strong Ag–O bonding and high phase purity. The synthesized nanoparticles exhibited notable catalytic activity, demonstrating their potential as efficient catalysts in environmental and chemical processes. Future work will focus on surface modification and the development of Ag_2O-based composite catalysts to further enhance catalytic efficiency, selectivity, and long-term stability.

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