Kongunadu Research Journal 12 (1): 55-61, 2025
Publisher: Kongunadu Arts and Science College, Coimbatore.

https://krjournal.com/index.php/krj/article/view/681

DOI:10.26524/krj.2025.9

RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF CHITOSAN BASED SILVER-OXIDE NANOPARTICLES

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ABSTRACT

Silver oxide nanoparticles (AgO NPs) have attracted significant interest due to their remarkable physicochemical properties, antimicrobial activity, and biocompatibility, making them ideal candidates for a variety of industrial, environmental, and biomedical applications. However, their practical utilization requires stabilization and biocompatibility, which can be achieved by integrating them with natural biopolymers like chitosan. Chitosan, a biodegradable and non-toxic polysaccharide, serves as an effective stabilizer and functionalizing agent, preventing nanoparticle aggregation and enhancing their biological activity. The nanocomposites were synthesized through an eco-friendly approach, with chitosan acting as both a reducing and capping agent. Characterization techniques, including UV, XRD, SEM with EDX, were employed to confirm the formation, size, and dispersion of the silver oxide nanoparticles. In conclusion, chitosan-based silver oxide nanoparticles represent versatile, eco-friendly materials with broad potential for applications. Further research is needed to optimize synthesis methods and enhance nanoparticle stability.

Keywords: Silver-oxide**,** Chitosan, Aggregation, Nanocomposites, Biocompatibility.

Introduction

Silver oxide nanoparticles (AgO NPs) have gained significant attention due to their remarkable catalytic, physicochemical properties, biocompatibility, and antimicrobial activity, making them valuable in various industrial, environmental, and biomedical applications. These nanoparticles exhibit unique physicochemical characteristics, such as high surface-to-volume ratio and exceptional reactivity, which contribute to their effectiveness in inhibiting microbial growth and promoting wound healing. However, the successful deployment of AgO nanoparticles in such domains requires stabilization and biocompatibility, which can be achieved by combining them with biopolymeric materials such as chitosan [1].

Chitin is a linear copolymer consisting of β-(1-4)-linked units of 2-amino-2-deoxy-D-glucan and 2-acetamido-2-deoxy-D-glucan. Structurally, it is composed of β-(1-4)-linked D-glucosamine units. Unlike cellulose, where the hydroxyl group is present, chitin has an N-acetyl group ($-NHCOCH_3$) substituting it. Chitin primarily exists in three polymorphic forms: α, β, and γ.Among these, α-chitin is the most abundant form [2]. The major source of α-form of chitin is generally shrimps, insect cuticle, crab, krill, lobster, cell wall of yeast and Zygomycetes. The abundancy of -chitin favors the significant quality of chitin as high crystallinity and purity due to the absence of calcium carbonate, proteins, and pigments. Instead β-chitin is found in connotation with proteins in squid pens while γ-chitin is found in cuttlefish stomach lining [3]. X-Ray diffraction revealed that the inner ring present in α-form of chitin is unaffected from hydration while the inner ring of β-chitin is sensitive to hydration. Moreover crystallographically, α-chitin exhibits two antiparallel molecules per unit cell, whereas β-chitin exhibits one parallel arrangement. As far as similarity is concerned, both the allomorphs have same moiety of N-acetylglycosamine [4].

METHODOLOGY

2.1 MATERIALS:

The materials used in this experiment include chitosan, a natural biopolymer known for its biocompatibility and biodegradability, which was dissolved in 10% acetic acid to form a chitosan solution. Silver Nitrate ($AgNO_3$) were used as metal precursors for the synthesis of metal oxide nanoparticles, Sodium hydroxide (NaOH) was employed to adjust the pH and precipitate metal hydroxides and purchased from Global chemicals, Hosur, Tamil Nadu. The reaction mixtures were prepared using standard laboratory glassware, including a beaker and standard measuring flask, to ensure accurate measurement of all solutions. A magnetic stirrer machine equipped with a stirrer bar was used continuously to stir the solutions, ensuring uniform mixing throughout the synthesis process.

2.2 METHODS:

2.2.1 Preparation of chitosan:

For chitosan to be extracted from dried mushroom it is necessary to convert it first to chitin. Generally, extraction of chitin from dried mushroom consists of three steps including demineralization for removal of calcium carbonate/phosphate, deproteinisation for removal of protein and then, chitin can be converted into chitosan by N-deacetylation which partially removes the acetyl group from the polymers chain composition. Mushroom was washed to remove the residue attached to it and then dried in intense sunlight and left for autolysis for 24 hours at room temperature which improve the quality of chitosan.[5]

2.2.2 Preparation of Chitosan / Silver Nitrate Oxide Nanoparticles:

Chitosan-based silver oxide (AgO) nanoparticles were prepared using a chemical co-precipitation method involving silver nitrate (AgNO3) and sodium hydroxide (NaOH). Initially, a 1% (w/v) chitosan solution was prepared by dissolving chitosan in 1% (w/v) acetic acid under constant stirring at room temperature until a clear solution was obtained. Separately, an aqueous solution of AgNO3 was prepared, which served as the silver precursor for AgO formation. This silver nitrate solution was slowly added to the chitosan solution under vigorous stirring to ensure uniform dispersion of copper ions within the chitosan matrix. To initiate the formation of AgO nanoparticles, NaOH solution was added dropwise to the $AgNO_3$-chitosan mixture, leading to the precipitation of silver hydroxide $Ag(OH)_2$. The reaction was allowed to proceed under continuous stirring with 600-700 rpm for about 18 hrs, Until the pH of the mixture reached around 9-10, at which point $Ag(OH)_2$ the blue color precipitated out. The mixture was then heated to convert $Ag(OH)_2$ into AgO nanoparticles. The resulting chitosan-AgO nanoparticle suspension was centrifuged to separate the nanoparticles, which were then washed with distilled water to remove any residual reactants.

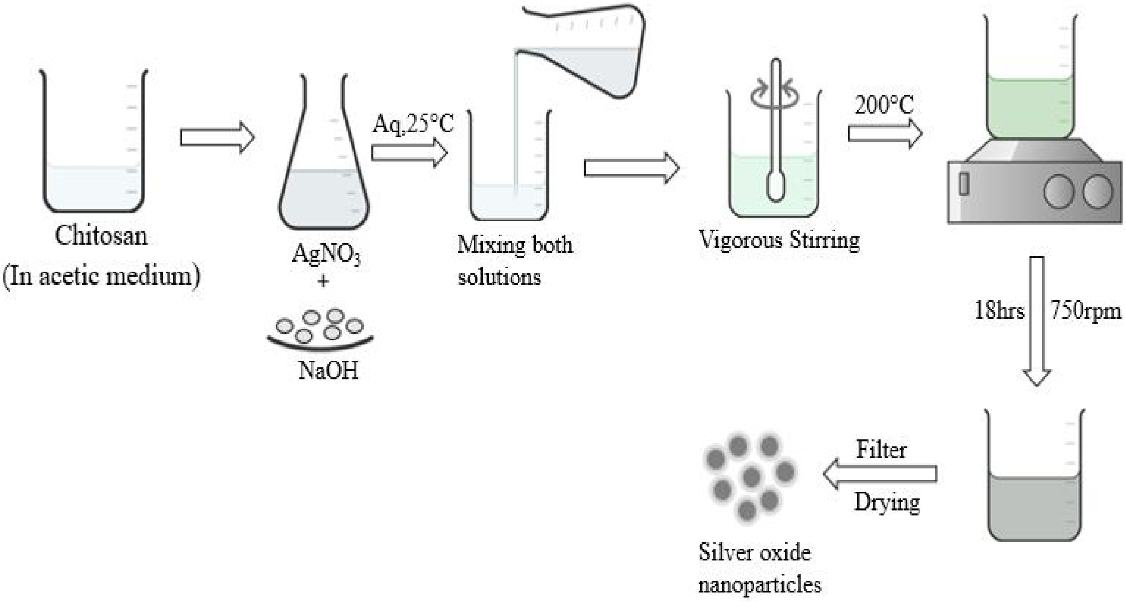


Figure.1 Preparation of chitosan based silver oxide nanoparticles

Reaction:

$$AgNO_3 + 2NaOH \xrightarrow{(i)} Ag(OH)_2 + Na_2NO_3$$

$$Ag(OH)_2 \xrightarrow{(ii)} AgO + H_2O$$

(i) Aqueous medium,25°C (ii) In air, 200°C - 300°C

RESULTS AND DISCUSSIONS:

3.1 Ultraviolet – Visible Spectroscopy (UV) Analysis:

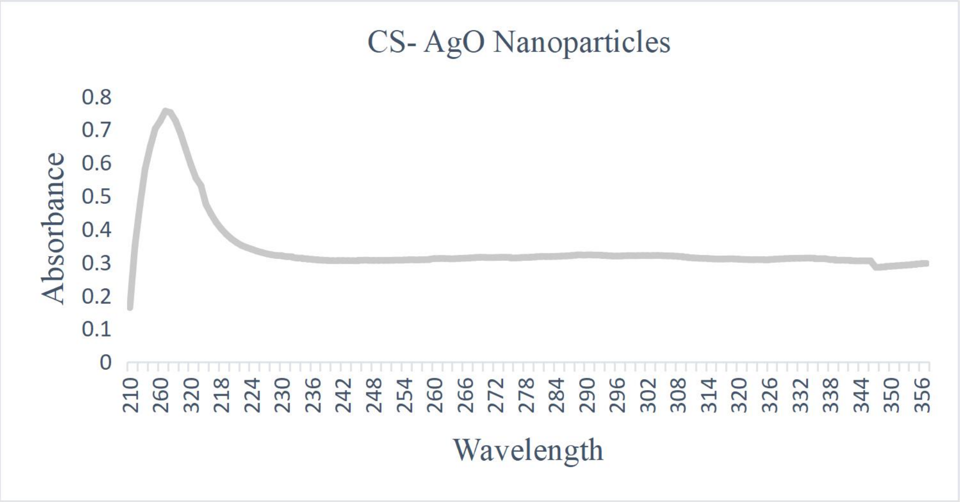


Figure.2 UV Spectra of Chitosan/AgO nanoparticles

Silver oxide nanoparticles exhibit a characteristic SPR absorption peak typically in the 300 nm range, which is primarily attributed to the excitation of surface electrons in the silver (Ag) component of the nanoparticles. The presence of chitosan often leads to narrowing of the SPR peak compared to unmodified AgO nanoparticles, indicating better stabilization and less aggregation. Impact of chitosan: The band gap of AgO nanoparticles may be slightly reduced in the presence of chitosan due to changes in the electron density and energy states at the nanoparticle surface, which enhances photocatalytic and antimicrobial activity.

3.2 Fourier Transform Infra-Red (IR) Analysis:

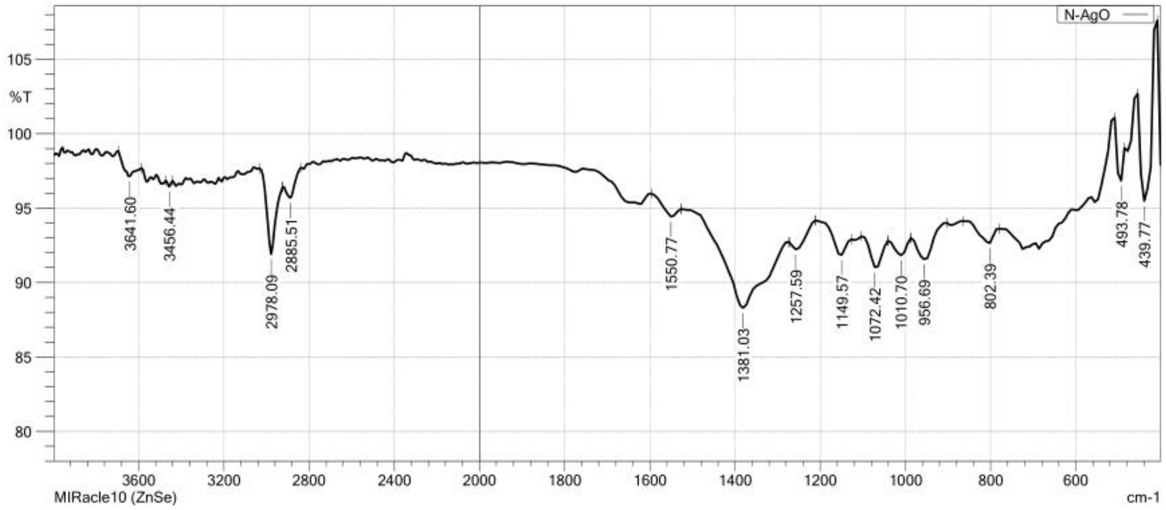


Figure.3 FTIR analysis of Chitosan/AgO

A broad absorption band 3456.44 cm^{-1} corresponds to the O-H and N-H stretching vibrations, indicating the presence of hydroxyl and amine groups in chitosan. The peak at 2885.51 cm^{-1} in the IR spectrum of chitosan typically corresponds to C-H stretching vibrations of the aliphatic $-CH_2$ and $-CH_3$ groups present in the chitosan backbone. This is associated with the saccharide structure of chitosan. The peak at 1550 cm^{-1} represents the amide II band (N-H bending) which is also C=O axial deformation band. The broad peak at 1381 cm^{-1} corresponds to CH_3 symmetrical deformation, confirming the structure of chitosan.

For AgO Nanoparticles, A band 439.77 cm^{-1} confirms the presence of Ag–O bonds. The interaction of AgO with chitosan may enhance or shift the absorption bands related to the amine or hydroxyl groups.

- The FTIR results confirms that the chitosan structure remains intact during the nanoparticle synthesis process, as the characteristic peaks of chitosan were preserved.
- The successful incorporation of ZnO, CuO, and AgO nanoparticles is evident from the appearance of characteristic metal-oxide peaks in the spectrum.
- The interactions between chitosan and metal oxides (ZnO, CuO, AgO) can be attributed to the formation of hydrogen bonds or electrostatic interactions between the amine, hydroxyl groups of chitosan, and the surface of the nanoparticles.

These interactions enhance the stability of the composite nanoparticles and potentially improve their antimicrobial, catalytic, or other functional properties.

3.3 X- RAY DIFFRACTION (XRD) ANALYSIS:

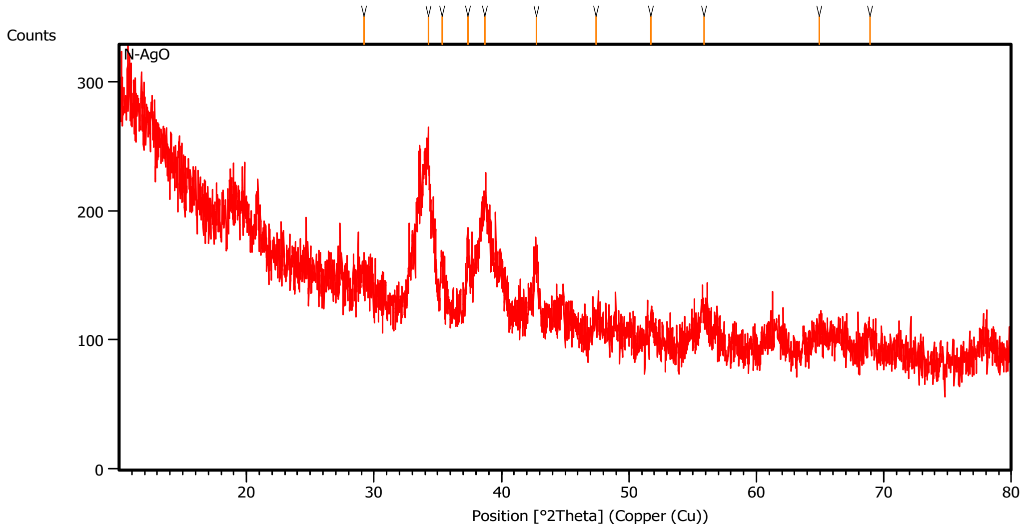


Figure.4 XRD Main graphics, Analyze view for AgO

2θ	FWHM	d-spacing [Å]	D	delta	Micro strain	Lattice parameters, a	Relative intensity
29.2301	0.8029	3.05535	8.63	13.41	6.260	157.74	12.31
34.263	0.2007	2.61719	32.71	0.93	1.285	136.822	100.00
35.3434	0.2007	2.53963	32.28	0.95	1.234	133.16	27.17
37.3549	0.2007	2.40737	31.46	1.01	1.147	126.95	37.38
38.6828	0.5353	2.32774	11.58	7.45	2.917	123.24	60.17
42.7214	0.3346	2.11658	17.44	3.28	1.580	113.54	38.41
47.4113	0.6691	1.91757	8.03	15.48	2.683	104.62	9.92
51.7458	0.4015	1.76668	12.25	6.66	1.381	98.09	16.11
55.87	0.5353	1.64565	8.32	14.42	1.5837	93.05	24.73
64.9486	0.8029	1.43585	4.18	56.97	1.637	85.02	12.34
68.9212	0.8029	1.36247	3.55	78.96	1.350	82.55	10.14

Table 4.3 Calculation of Crystallite Size, Microstrain, Lattice parameters of AgO

For AgO- Chitosan Nanoparticles,

(i) Peak Positions (2θ):

- The characteristic peaks of AgO are observed at: 29.2°,*34.2°,35.3°,37.3°*,38.6°
- For AgO at 2θ=34.2° which is corresponding to the (100) plane,
- The inter planar spacing d is approximately 0.261 nm.
- The X-ray wavelength λ is approximately 0.1563 nm (or 1.563 Å).

(ii) Crystallite Size:

The crystallite size of AgO nanoparticles in chitosan composites ranges of 32 nm.

(iii) Chitosan Interaction:

The incorporation of chitosan often leads to peak broadening and reduced intensity, indicating effective dispersion and reduced crystallinity of AgO.

(iv) Structure:

Silver oxide (AgO) has a monoclinic crystal structure, belonging to the $P2_1/c$ space group AgO in nanoparticle form shows peak broadening in XRD due to reduced particle size and Lattice strain, allowing estimation of crystallite size via the Debye-Scherer equation.

This structure makes AgO an interesting material for applications like catalysis, batteries, and sensors.

3.4 SEM WITH EDAX:

3.4.1 Sem Analysis

(i) Morphology: SEM images of chitosan-AgO nanoparticles predominantly spherical or quasi-spherical shapes. Silver oxide (AgO) nanoparticles often exhibit a more uniform and smaller size due to the strong stabilizing effect of chitosan.

(ii) Particle Size: The SEM images typically show sizes of 23.68nm, 25.01nm, 23.93nm, 19.40nm, 23.58nm and 31.44nm with well-dispersed particles, with a tendency to form more compact clusters. Chitosan prevents excessive agglomeration, keeping the particles well-separated.

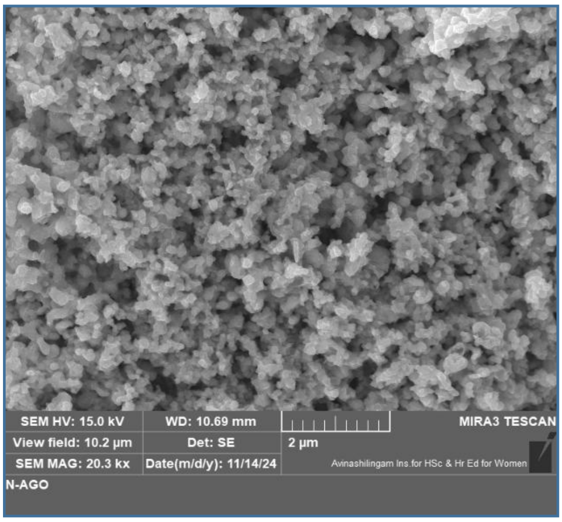
(iii) Dispersion: The chitosan matrix effectively prevents agglomeration of AgO particles. SEM images show a high degree of uniformity and well-separated nanoparticles.

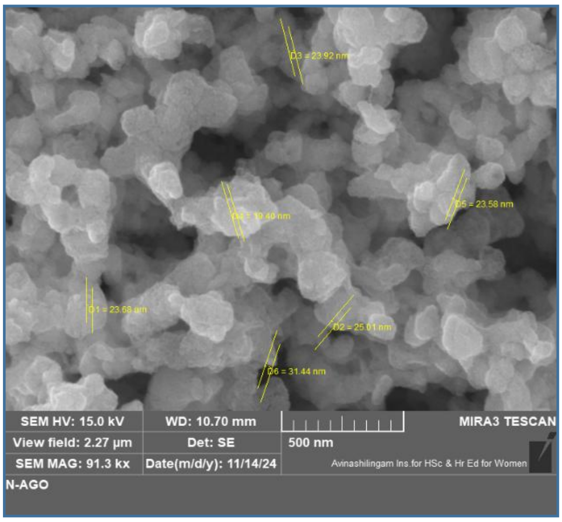
(iv) Surface Texture: The surfaces of AgO nanoparticles are typically smooth, but slight irregularities may be visible due to the AgO crystal structure. Chitosan-coated AgO nanoparticles show a thin, uniform layer of chitosan surrounding the metal oxide core.

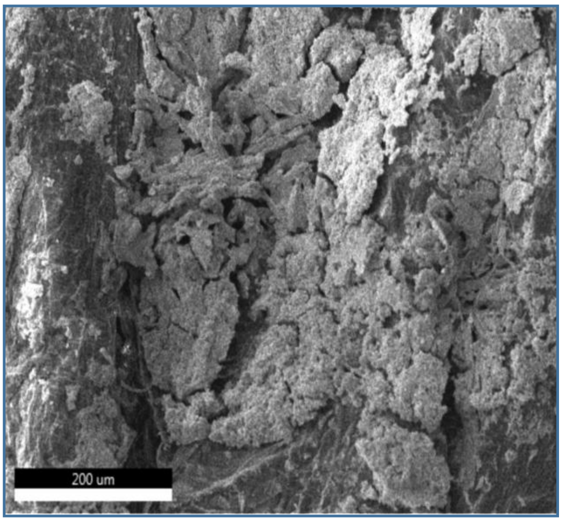
3.4.2 EDAX Analysis:

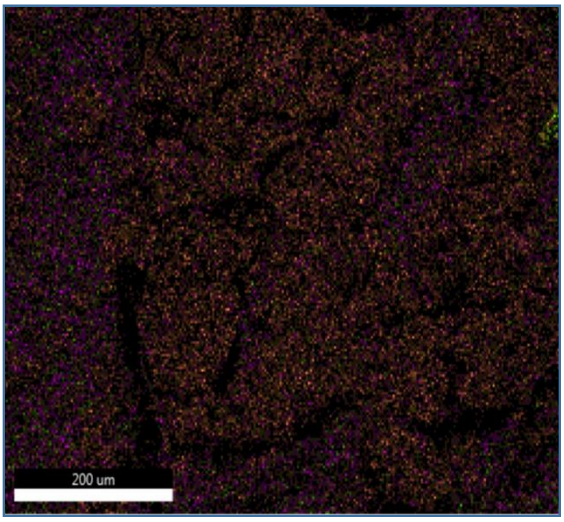
(i) Composition: The EDAX spectrum confirms the presence of silver (Ag) and oxygen (O), along with carbon (C) and nitrogen (N) from chitosan.

(ii) Elemental Ratios: The Ag-to-O ratio is consistent with the expected stoichiometry of AgO. The C and N peaks confirm the chitosan coating around the nanoparticles.









🟪 19% C K
🟩 15% O K
🟫 6% NaK
🟧 61% AgL

Element	Weight%	Atomic%
C K	9.02	26.28
O K	21.12	46.20
Na K	4.05	6.17
Ag L	65.81	21.35

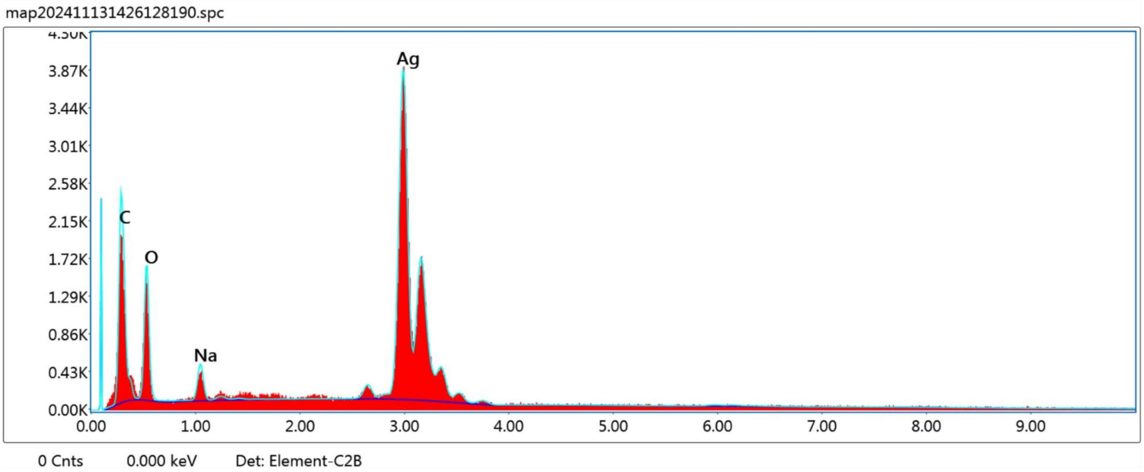


Figure.5 SEM and EDAX images with different magnifications for AgO nanoparticles

3.4.3 General Discussion for SEM-EDAX Results:

(i) Morphology and Particle Size:

- The morphology and particle size variation are influenced by synthesis parameters such as precursor concentration, pH, temperature, and chitosan concentration.
- SEM images confirm that ZnO, CuO, and AgO nanoparticles exhibit distinct morphologies—spherical (ZnO, AgO) and irregular or flake-like (CuO)—influenced by chitosan.
- ZnO and CuO nanoparticles show slightly larger particle sizes compared to AgO due to differences in crystal growth mechanisms and precursor reactivity.

(ii) Role of Chitosan:

- Chitosan stabilizes the nanoparticles, preventing aggregation , ensuring uniform size dispersion and reduced agglomeration.
- This is evident from the smooth coating seen in SEM and the carbon and nitrogen signals detected in EDX.
- The interaction of chitosan with metal ions (Zn^{2+}, Cu^{2+}, Ag^{+}) through hydroxyl and amine groups helps in nanoparticle stabilization.

(iii) Elemental Composition:

- EDAX analysis confirms the presence of Zn, Cu, and Ag along with oxygen, validating the synthesis of the respective nanoparticles.The C and N peaks in all spectra confirm the successful encapsulation of nanoparticles by the chitosan matrix.

(iv) Surface Characteristics

- The encapsulation of nanoparticles by chitosan results in smooth surfaces for ZnO and AgO, while CuO exhibits some roughness due to intrinsic material properties.
- SEM images show evidence of a thin chitosan layer, confirming its role as a biopolymer coating.

(v) Aggregation Behavior

- Aggregation is minimal due to the electrostatic stabilization provided by chitosan, as observed in the SEM images.
- Without chitosan, bare nanoparticles tend to cluster, reducing their effectiveness in applications.

CONCLUSION:

In this investigation, the synthesis, characterization of chitosan-based silver oxide nanoparticles (AgO NPs) have been systematically studied. The use of chitosan as a biocompatible and eco-friendly stabilizer has proven to be effective in controlling the size, shape, and dispersion of silver oxide nanoparticles, which significantly enhances their stability and functional properties. The interaction between chitosan and silver ions leads to the formation of highly stable silver oxide nanoparticles with well-defined characteristics.In conclusion, the successful synthesis and application of chitosan-based silver oxide nanoparticles offer a pathway for developing highly functional, eco-friendly nanomaterials with a wide array of practical uses. Their unique properties, including antimicrobial activity, photocatalytic potential, and biocompatibility, position them as valuable candidates for future research and technological advancements.

ACKNOWLEDGEMENT

I gratefully acknowledge the contributions to professor Dr.J.Johncy caroline for their insightful guidance and continuous support throughout the research process. I would like to express my sincere gratitude to the management of Nirmala College For Women, to their constant support to access their laboratories. The green synthesis process of this research work is done to help in environmental conditions.

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