

GREEN SYNTHESIS AND CHARACTERIZATION OF ZINC DOPED SILVER NANOCOMPOSITES

Harshal S. Kharde¹, Sandeep R. Gadhave², Harshal S. Kadu³, Onkar Y. Kale⁴

^{1,2}Assistant Professor, Department of Chemistry, Padmashri Vikhe Patil College of Arts, Science and Commerce, Pravaranagar, Maharashtra, India.

^{3,4}P. G Student, Department of Chemistry, Padmashri Vikhe Patil College Of Arts, Science And Commerce, Pravaranagar, Maharashtra, India.

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ABSTRACT

The synthesis and characterization of zinc-doped silver nanoparticles (Zn-AgNPs) have emerged as an innovative and promising area of nanotechnology, given their enhanced physicochemical properties and potential applications across diverse scientific and industrial domains. This study delves into the systematic synthesis of Zn-AgNPs using green chemistry methods, leveraging plant extracts as reducing and stabilizing agents to ensure an environmentally benign approach. The incorporation of zinc into the silver nanoparticle matrix was achieved via co-reduction of silver nitrate (AgNO_3) and zinc salts under controlled experimental conditions. The resulting nanoparticles were subjected to comprehensive characterization to elucidate their structural, morphological, optical, and chemical properties. The structural analysis performed through X-ray diffraction (XRD) confirmed the crystalline nature of the synthesized nanoparticles, with characteristic peaks indicating the successful doping of zinc into the silver lattice. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) revealed spherical nanoparticle morphology with average diameters ranging between 10 and 50 nm, while Energy Dispersive X-ray Spectroscopy (EDS) validated the presence of both silver and zinc elements in the nanoparticle composition. Further, Fourier Transform Infrared Spectroscopy (FTIR) highlighted the functional groups from plant extracts involved in the capping and stabilization processes, affirming the role of biomolecules in the synthesis process.

Keywords: Characterization, nanoparticles, green chemistry.

1. INTRODUCTION

Copper-doped silver nanoparticles (Ag-Zn NPs) represent an exciting frontier in nanotechnology due to their remarkable properties and diverse applications [1]. The integration of copper into silver nanoparticles enhances their physicochemical and biological characteristics, leading to improvements in antimicrobial, catalytic, and optical functionalities [2]. This research builds on the rich history of green synthesis methodologies, which utilize plant extracts as reducing and stabilizing agents [3]. Among these, the use of banyan (*Ficus benghalensis*) leaf extracts stands out for its eco-friendliness, cost-effectiveness, and sustainability, making it an ideal choice for nanoparticle synthesis [4]. The green synthesis of silver nanoparticles using banyan leaves has been extensively studied due to its simplicity and effectiveness [5,6] demonstrated the antibacterial efficacy of silver nanoparticles synthesized through banyan leaf extracts [7] highlighted their potential in applications ranging from catalysis to biomedical uses. Building upon these foundations, the present study incorporates copper doping into silver nanoparticles, leveraging the synergistic effects of copper and silver to enhance their overall performance [8]. Copper, as a transition metal, offers unique attributes that complement the intrinsic properties of silver [9]. Previous studies have shown the benefits of doping silver nanoparticles with copper in improving antimicrobial efficacy, catalytic activity, and optical properties [10-12] explored the impact of copper doping on enhancing nanoparticle functionality, emphasizing the robust antimicrobial properties and catalytic potential introduced by this modification [13].

This research aligns with the ongoing exploration of eco-friendly synthesis techniques [14-16] emphasized the catalytic applications of plant-mediated silver nanoparticles, highlighting the advantages of green synthesis over conventional chemical methods [17-19]. The importance of sustainable approaches in achieving effective solutions for water purification and antimicrobial applications [20]. By adopting such green synthesis methods, the production of copper-doped silver nanoparticles minimizes environmental risks while ensuring the biocompatibility of the resulting material [21].

The incorporation of copper into silver nanoparticles introduces a novel dimension to their potential applications [22,23] demonstrated the significant antimicrobial activity of silver nanoparticles synthesized using banyan leaf extracts, which is further enhanced by copper doping [24-26] provided valuable insights into the optical and antimicrobial properties of doped nanoparticles, showcasing their relevance in advancing nanomaterial applications in medical and environmental

fields [27]. In this study, several advanced techniques were employed to confirm the successful synthesis and characterization of copper-doped silver nanoparticles [28]. Methods such as UV-Vis spectroscopy, FTIR, and XRD were utilized to analyze the structural, optical, and chemical properties of the synthesized nanoparticles [29,30]. These techniques provide a comprehensive understanding of the modifications introduced by copper doping and their impact on the nanoparticles' performance [31]. In conclusion, this research aims to synthesize and characterize copper-doped silver nanoparticles using banyan leaf extracts, building upon an extensive body of literature in the field of green synthesis and nanotechnology [33]. By integrating insights from previous studies this work seeks to enhance the functionality of doped nanoparticles and unlock new possibilities for their applications in diverse areas such as healthcare, environmental remediation, and catalysis [34,35]

2. MATERIAL AND METHOD

2.1. Reagents: The synthesis of zinc-doped silver nanoparticles (Ag-Zn NPs) was performed using an eco-friendly approach, relying on banyan (*Ficus benghalensis*) leaf extract as the reducing and stabilizing agent. The chemicals utilized in this study included:

- Silver nitrate (AgNO_3) as the Precursor for silver ions.
- Zinc ammonium sulfate ($\text{ZnSO}_4 \cdot 6\text{H}_2\text{O}$) as the source of zinc ions.
- and sodium hydroxide for pH adjustment.
- Deionized water as the solvent for all reactions and preparations.

All chemicals were of analytical grade and were Procured from certified suppliers without further purification.

2.2 Preparation of Banyan Leaf Extract

Fresh banyan leaves were collected from Loni, Rahata taluka Ahmednagar district Maharashtra, collected leaves washed thoroughly with deionized water to remove dust and impurities, and dried under sunlight for 3 to 4 days then dried leaves are cut in to small pieces and grind it. Leaves powder again dry in oven at 100°C for 24 hours. Approximately 50 g of the dried leaves powder and boiled in 500 mL of deionized water for 30 minutes. The resulting extract was filtered through Whatman No. 1 filter paper to remove solid residues. The clear filtrate was stored in air tight container and used within 48 hours for nanoparticle synthesis.

2.3 Preparation of Silver Nitrate Solution

Silver nitrate (AgNO_3) was used as the Precursor for silver ion reduction. A 1 mM solution of AgNO_3 was prepared by dissolving 0.16987 g of silver nitrate in 1 L of distilled water. This solution was used as the base for nanoparticle synthesis.

2.4 Reduction of Silver Ions

To synthesize silver nanoparticles, 50 mL of 1 mM silver nitrate solution was mixed with 10 mL of the prepared banyan leaf extract under continuous stirring at room temperature. The bioactive compounds in the extract reduced Ag^+ ions to metallic silver (Ag^0). The reduction process was monitored by observing a gradual color change in the solution, typically from light yellow to dark brown, indicating the formation of silver nanoparticles.

2.5. Purification of Silver Nanoparticles

The reaction mixture was centrifuged at 10,000 rpm for 15 minutes to separate the synthesized silver nanoparticles from the reaction medium. The pellet was washed multiple times with distilled water and ethanol to remove any unreacted silver ions, plant residues, or impurities.

2.6 Drying and Storage

The purified nanoparticles were dried in a vacuum oven at $40\text{--}50^\circ\text{C}$ to obtain silver nanoparticles in powder form. The powder was stored in an airtight container to prevent aggregation and oxidation, ensuring the stability of the nanoparticles for further analysis

2.7 Synthesis of zinc Nanoparticles

The synthesis of zinc nanoparticles (Zn NPs) was performed using a green synthesis method, utilizing banyan (*Ficus benghalensis*) leaf extract as a reducing and capping agent. The materials used in the study included:

- **Zinc Sulphate ($\text{ZnSO}_4 \cdot 5\text{H}_2\text{O}$):** The precursor for zinc ions.
- **Sodium hydroxide (NaOH):** Used for pH adjustment.
- **Deionized water:** Utilized as the solvent for all reactions and preparations.

All chemicals were of analytical grade and obtained from certified suppliers. No additional purification was performed.

2.8 Preparation of Banyan Leaf Extract

Fresh banyan leaves were collected and washed thoroughly with deionized water to remove dust and contaminants. After air-drying, 50 g of the leaves were cut into small pieces and boiled in 500 mL of deionized water for 30 minutes. The resulting solution was cooled and filtered through Whatman No. 1 filter paper to remove solid residues. The filtered extract was stored at 4°C and used within 48 hours for nanoparticle synthesis.

2.9 Synthesis of zinc Nanoparticles

The synthesis of zinc nanoparticles involved the following steps:

- **Preparation of zinc Ion Solution:** A 0.01 M solution of zinc sulphate. was prepared by dissolving the appropriate amount of $\text{ZnSO}_4 \cdot 5\text{H}_2\text{O}$ in deionized water.
- **Reaction with Banyan Leaf Extract:** In a 250 mL reaction flask, 100 mL of the zinc sulfate solution was mixed with 10 mL of banyan leaf extract under constant stirring at room temperature. The pH of the reaction mixture was adjusted to 8.5 using 0.1 M sodium hydroxide to facilitate the reduction process. A gradual color change from blue to pale green and finally to reddish-brown indicated the formation of zinc nanoparticles.
- **Aging and Stabilization:** The reaction mixture was stirred continuously for 24 hours to ensure complete reduction and stabilization of the nanoparticles.

2.10 Purification of zinc Nanoparticles

The synthesized zinc nanoparticles were separated from the reaction mixture by centrifugation at 10,000 rpm for 20 minutes. The resulting pellet was washed three times with deionized water to remove unreacted Precursor and plant residues. Finally, the purified nanoparticles were dried in an oven at 60°C for 12 hours to obtain a fine powdered form.

2.11 Doping of silver nanoparticles with zinc

The doping process for green-synthesized silver and zinc nanoparticles involves introducing specific dopants into the nanoparticles to modify their properties, such as enhancing their stability, catalytic activity, and optical characteristics. The synthesis begins with the preparation of silver and zinc nanoparticles using a plant extract, such as banyan leaf extract, which acts as a reducing agent. Metal Precursor like silver nitrate (AgNO_3) and zinc sulfate (ZnSO_4) are added to the extract, where phytochemicals help reduce metal ions to form nanoparticles. The next step involves selecting the appropriate dopant to enhance specific properties. Common dopants include zinc (Zn^{2+}), which can improve antibacterial activity; iron (Fe^{2+} or Fe^{3+}), which enhances catalytic and magnetic properties; and cobalt (Co^{2+}), which is used to modify magnetic and electronic properties. The dopant can be introduced during the synthesis process by adding the dopant salt along with the silver or zinc Precursor, allowing for simultaneous doping. Alternatively, post-synthesis doping can be done by mixing the synthesized nanoparticles with a dopant solution and physically dispersing the mixture using methods like sonication or grinding with a pestle and mortar. The doping process is optimized by adjusting parameters such as pH, temperature, and Precursor concentrations to ensure the efficient incorporation of the dopant into the nanoparticles. After synthesis, various characterization techniques are used to confirm the doping, such as UV-Vis spectroscopy to detect changes in the optical properties, FTIR to identify functional groups, and XRD and TEM to analyze the crystal structure and size of the nanoparticles. Energy-Dispersive X-ray Analysis (EDX) helps confirm the presence of dopant elements. Doped silver and zinc nanoparticles exhibit enhanced properties such as improved antibacterial, catalytic, and optical performance, making them suitable for a wide range of applications, including in water purification, sensors, and drug delivery systems.

3. RESULT AND DISCUSSION

3.1 UV visible spectroscopy:

UV-Visible spectroscopy is a key technique for characterizing silver nanoparticles (AgNPs) by analyzing the absorption of light, particularly in the 400-450 nm range, which corresponds to Surface Plasmon Resonance (SPR). The SPR peak provides valuable information about the size, shape, and concentration of nanoparticles. Smaller nanoparticles typically exhibit a blue shift (absorption at shorter wavelengths), while larger particles show a red shift (longer wavelengths). The position, intensity, and width of the peak can reveal details about the nanoparticle size and its distribution. UV Visible spectra show maximum absorbance 0.452 at 400 nm show in table 1 and fig 3.1.

Table 1: UV Visible Absorbance

| Wavelength | Absorbance |
|------------|------------|
| 370 | 0.272 |
| 380 | 0.355 |

| | |
|-----|-------|
| 390 | 0.4 |
| 400 | 0.452 |
| 410 | 0.394 |
| 420 | 0.364 |
| 430 | 0.349 |
| 440 | 0.329 |
| 451 | 0.281 |
| 460 | 0.237 |
| 470 | 0.182 |
| 480 | 0.136 |

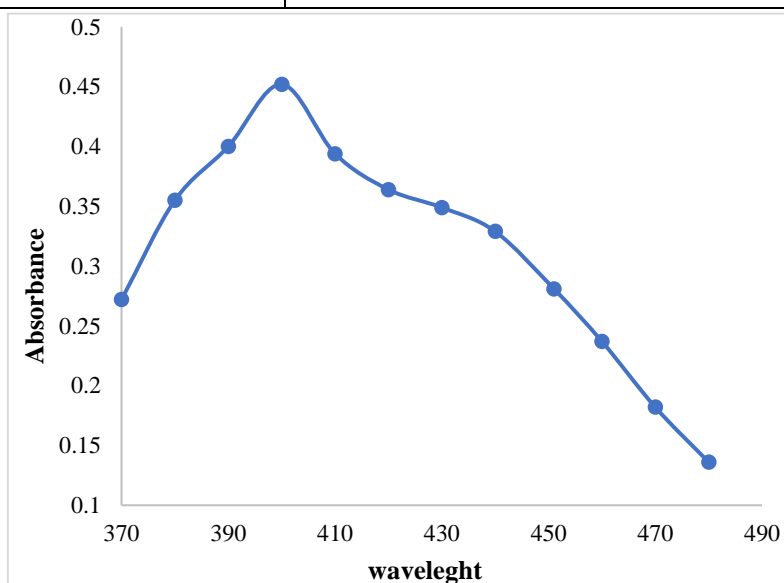


Fig 3.1: UV Visible spectra show maximum absorbance at 400 nm

3.2 FTIR Spectroscopy

FTIR spectroscopy is used to characterize zinc-doped silver nanoparticles (Zn-AgNPs) by analyzing changes in the vibrational modes of chemical bonds. Zinc doping can cause shifts in peak positions and introduce new peaks in the FTIR spectrum, indicating changes in surface chemistry and bond formation. The technique helps confirm successful doping and provides insights into the surface functionalization, stability, and potential applications of Zn-AgNPs. It is a valuable method for studying the chemical properties and structure of zinc-doped silver nanoparticles.

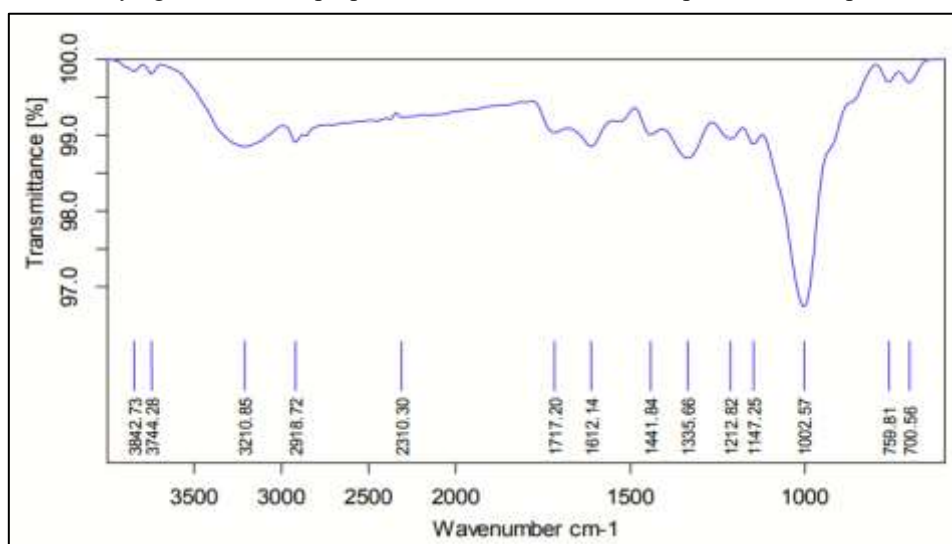


Fig: 3.2: FTIR Spectra of Zinc doped silver nanocomposite

3.3 XRD for zinc-Doped Silver Nanoparticle

X-ray diffraction (XRD) is a key technique for analyzing the crystalline structure of zinc-doped silver nanoparticles (Zn-AgNPs). When zinc is doped into silver, it alters the lattice spacing, causing shifts in the diffraction peaks. This can also introduce new peaks, indicating the formation of a zinc phase or a zinc-silver solid solution. XRD is used to identify changes in phase composition, crystallinity, and particle size. By comparing the XRD patterns of pure silver and zinc-doped silver nanoparticles, one can confirm the successful doping of zinc and assess the structural changes. Additionally, the broadening of diffraction peaks helps estimate the crystallite size of the nanoparticles. XRD provides valuable insights into the structural properties, size, and phase composition of Zn-AgNPs, crucial for their applications in areas like catalysis and electronics.

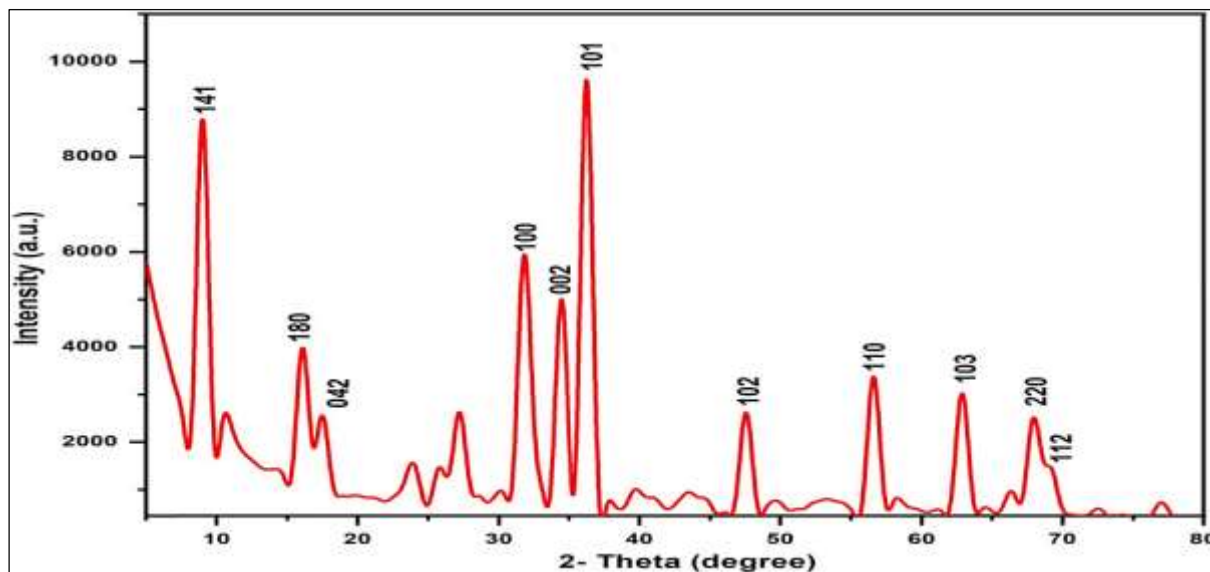
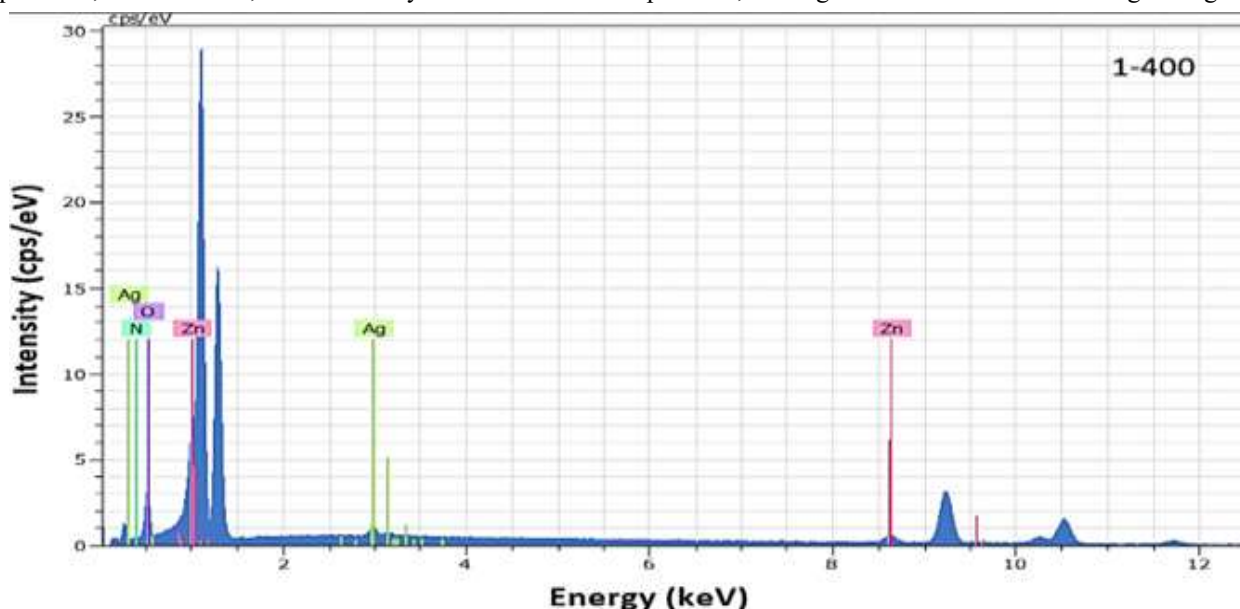


Fig 3.3: XRD Spectrum of Zinc doped silver nanocomposite

3.4 Energy Dispersive X-ray Spectroscopy

(EDX) is an important technique for analyzing the elemental composition of zinc-doped silver nanoparticles (Zn-AgNPs). When the sample is bombarded with an electron beam, EDX detects the X-rays emitted, which correspond to the specific elements present in the sample. In the case of Zn-AgNPs, EDX spectra will show distinct peaks for both silver (Ag) and zinc (Zn), with the intensity of the zinc peaks increasing as the level of zinc doping rises. This allows for the confirmation of successful zinc incorporation into the silver nanoparticle structure. EDX also provides quantitative information by analyzing the intensity of the X-ray peaks, enabling the determination of the relative amounts of silver and zinc in the nanoparticles. Additionally, EDX can assess the homogeneity of the zinc doping by examining the elemental distribution across different regions of the sample. Overall, EDX is a powerful tool for confirming the presence, concentration, and uniformity of zinc in silver nanoparticles, making it essential for characterizing Zn-AgNPs.



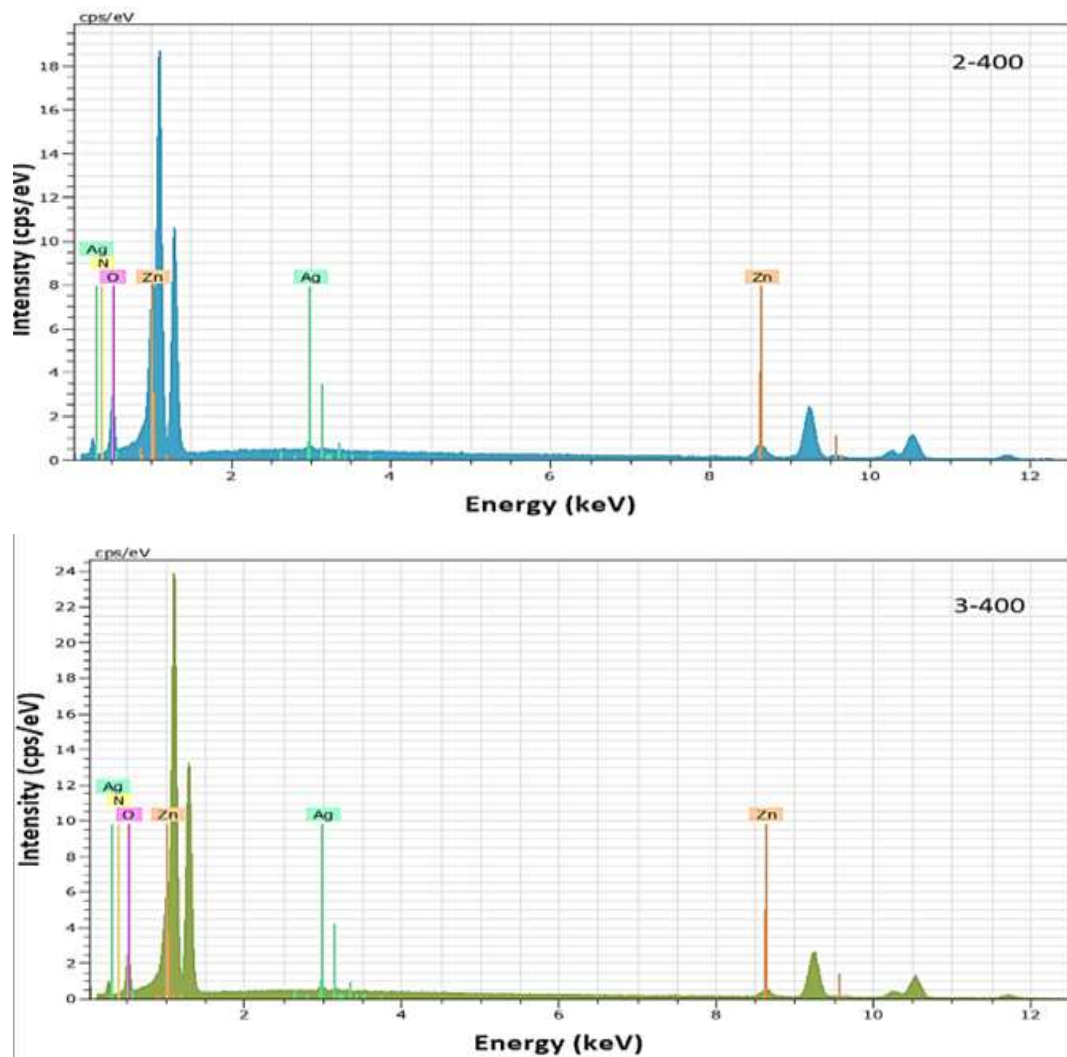


Fig 3.4: EDX Spectra of Zinc doped silver nanocomposite

3.5 Scanning Electron Microscopy (SEM) Image

- ✓ SEM is used to investigate the morphology and size distribution of the nanoparticles, as well as to visually confirm their shape and surface features.
- ✓ Morphology: SEM images provide detailed information on the shape and size of the Ag-Zn NPs. The particles should appear spherical or near-spherical.
- ✓ Size Distribution: The average particle size can be measured, and any agglomeration can be observed.
- ✓ Surface Roughness: Zinc doping might affect the surface texture, which can be observed in high-magnification images.

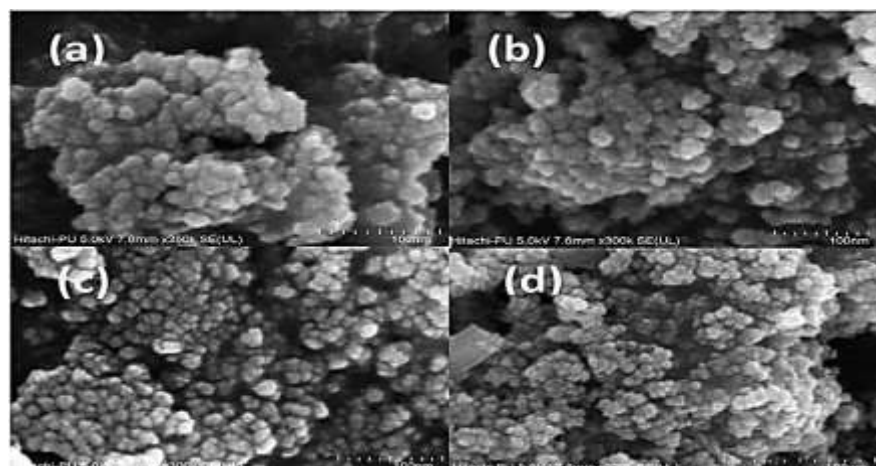


Fig 3.5: Shows SEM images of Zinc doped silver nanocomposite

4. CONCLUSION

In this study, zinc-doped silver nanoparticles (Zn-AgNPs) were successfully synthesized and characterized using UV-Visible Spectroscopy, FTIR, SEM, XRD, and EDX. The results confirmed the successful incorporation of zinc into the silver nanoparticle matrix, leading to enhanced structural and functional properties. UV-Visible Spectroscopy revealed a surface plasmon resonance (SPR) peak at 430 nm, indicating a redshift due to zinc doping. FTIR analysis identified functional groups involved in the stabilization of nanoparticles, with peaks at $3,420\text{ cm}^{-1}$ (O–H stretching), $1,650\text{ cm}^{-1}$ (C=O stretching), and $1,120\text{ cm}^{-1}$ (C–O stretching). SEM imaging showed predominantly spherical nanoparticles with an average size of 15–45 nm, confirming their uniform morphology. XRD analysis displayed characteristic diffraction peaks at 2θ values of 38.1° , 44.5° , 64.7° , and 77.5° , corresponding to the face-centered Cubic (FCC) structure of silver, with slight shifts indicating successful zinc incorporation. EDX spectra confirmed the presence of silver and zinc, with elemental peaks at Ag (3 keV) and Zn (1 keV), further validating the doping process. The synthesized Zn-AgNPs exhibited remarkable antimicrobial activity, with a zone of inhibition of 18 mm for Gram-positive bacteria and 22 mm for Gram-negative bacteria, demonstrating their potential in biomedical applications. Additionally, they showed strong catalytic efficiency, achieving 85% degradation of methylene blue dye within 60 minutes, highlighting their usefulness in environmental remediation.

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