



Microstructure-Modulated Antibacterial Performance of Chemically Precipitated SnO₂ Nanoparticles

Shatha Sh. Batros, Mohammed H. Ali, Ali J. Addie*

Center of Advanced Materials, Materials Research Directorate, Ministry of Science and Technology – Iraq

Article information

Article history:

Received: September, 05, 2023

Accepted: October, 20, 2023

Available online: December, 10, 2023

Keywords:

Tin oxide nanoparticles,
Antibacterial activity,
Chemical Precipitation,
Crystallite size control

*Corresponding Author:

Ali J. Addie

ali.jaddie@yahoo.com

Abstract

Tin oxide (SnO₂) nanoparticles were synthesized via a facile chemical precipitation route using tin chloride (SnCl₂•2H₂O) as precursor and ammonia as precipitant. The as-synthesized nanoparticles were subjected to post-calcination at 300°C, 400°C and 500°C and thoroughly characterized by advanced techniques including X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS) and Fourier transform infrared (FTIR) spectroscopy. XRD patterns revealed the formation of tetragonal SnO₂ crystalline phase with average crystallite sizes of 11.9 nm, 13.9 nm and 17.2 nm for the samples calcined at 300°C, 400°C and 500°C respectively. SEM micrographs demonstrated agglomerated and irregular morphology of the calcined SnO₂ nanoparticles. FTIR spectra confirmed the presence of characteristic Sn-O and O-Sn-O vibrational modes in the calcined SnO₂ samples. The antibacterial activity of the synthesized nanoparticles was evaluated against model Gram-negative (*Escherichia coli*) and Gram-positive (*Staphylococcus aureus*) bacterial strains by standard zone of inhibition assays. Remarkably, the SnO₂ nanoparticles exhibited excellent antibacterial activity due to their high specific surface area. A systematic increase in the inhibition zone diameter was observed with decrease in crystallite size of SnO₂ for both bacterial strains, suggesting an inverse relationship between crystallite size and antibacterial behavior. The present work demonstrates a simple, eco-friendly synthesis of antibacterial SnO₂ nanoparticles with controlled crystallite size by tuning the calcination temperature.

DOI: [10.53293/jasn.2023.7107.1246](https://doi.org/10.53293/jasn.2023.7107.1246), Department of Applied Sciences, University of Technology

This is an open access article under the CC BY 4.0 License.

1. Introduction

Tin oxide (SnO₂) nanoparticles have attracted considerable research interest in recent years owing to their versatile applications in gas sensing, optoelectronics, dye-based solar cells, photocatalysis, antibacterial agents, and other areas [1–4]. SnO₂ is an important n-type wide bandgap (E_g ~3.6 eV) semiconductor metal oxide characterized by its tetragonal rutile structure, excellent chemical stability, thermal stability up to 1100°C in air, high electron mobility, and good optical transparency in the visible region [5–8]. These remarkable properties are strongly influenced by the nanoparticle size, morphology, crystallinity, and surface characteristics.

Therefore, controlled synthesis of SnO₂ nanoparticles with tailored structural and optical properties is vital to fully exploit their applications across diverse technological fields. A variety of physical and chemical methods have been developed to synthesize SnO₂ nanoparticles including thermal evaporation [9], sol-gel [10], hydrothermal [11], microwave irradiation [12], chemical precipitation [13], electrochemical routes [14], etc. However, many of these techniques involve use of toxic and hazardous chemicals, high temperatures or high pressure conditions. There is need for facile, eco-friendly green chemistry approaches to produce high quality SnO₂ nanomaterials under ambient conditions. Wet chemical techniques like precipitation are attractive owing to their simplicity, low cost, and easy scaled up synthesis without need for sophisticated instrumentation [12, 15, 16]. Tin(II) oxide (SnO) is a useful precursor for controlled conversion to SnO₂ nanoparticles but its growth is challenging since Sn²⁺ tends to get oxidized to Sn⁴⁺ in air [17]. The morphological features including particle size, shape, porosity and crystallite size along with the bandgap and surface defects can be effectively tailored by tuning key synthesis parameters such as pH, aging time, temperature, etc [18, 19]. Calcination of the as-prepared SnO₂ precursor is commonly performed to improve the crystallinity and remove residual organics or moisture. The calcination temperature is found to significantly influence the nanoparticle morphology and crystallite growth [15, 20]. Inorganic nanomaterials exhibit higher thermal and chemical stability compared to organic antibiotics, making metal oxide nanoparticles such as SnO₂ very attractive antibacterial agents [15, 18, 21–23]. The antibacterial activity arises from the generation of reactive oxygen species on the nanoparticle surface and interactions with the cell membrane. The activity is dependent on the bacterial strain owing to differences in cell wall structure between Gram-positive and Gram-negative species [22,24]. Gram-positive bacteria have a thick peptidoglycan layer containing teichoic and lipoteichoic acids, while Gram-negative bacteria possess a thin peptidoglycan layer and an outer membrane containing lipopolysaccharides and membrane proteins. There is considerable interest in the application of SnO₂ nanoparticles as antibacterial agents against both types of pathogenic strains [17, 20]. However, systematic studies directly correlating the SnO₂ crystallite size, nanoparticle microstructure and surface defects with the antibacterial behaviour are still lacking. Despite the tremendous potential of SnO₂ nanoparticles as antibacterial agents, most prior works have utilized SnO₂ synthesized by methods involving toxic chemicals and harsh conditions [25–27]. Some biochemical techniques have been reported for SnO₂ synthesis but face challenges for scaled-up production [28, 29]. Furthermore, systematic correlations elucidating the dependence of antibacterial activity on the SnO₂ crystallite size, nanoparticle morphology, and surface defects are lacking. Hence, there is an urgent need to develop green chemistry approaches for controlled SnO₂ nanoparticle synthesis and establish structure-property relationships guiding their antibacterial applications [30, 31]. In this work, we seek to address this need for green synthesis and structure-property understanding of SnO₂ nanoparticles by developing an eco-friendly chemical precipitation route for tailored nanoparticle synthesis at room temperature. We utilize tin(II) chloride and ammonia precursors to synthesize SnO₂ nanoparticles with controlled crystallite size, phase purity, and surface defects by adjusting the precursor ratios and post-calcination temperatures. Additionally, we aim to provide new insights into the dependence of reactive oxygen species generation and antibacterial activity on the SnO₂ crystallite size and surface defects against model *Escherichia coli* and *Staphylococcus aureus* strains. The SnO₂ nanoparticles are thoroughly characterized by powder X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy dispersive x-ray spectroscopy (EDS) techniques. Furthermore, the antibacterial activity was evaluated against model Gram-negative *Escherichia coli* (*E. coli*) and Gram-positive *Staphylococcus aureus* (*S. aureus*) strains by standard zone of inhibition studies. This synthesis approach and correlation study provides a useful foundation for potential integration of green synthesized SnO₂ nanomaterials in water treatment and food packaging applications needing targeted antibacterial function.

2. Experimental

2.1. Materials

Tin(II) chloride dihydrate (SnCl₂·2H₂O, 99.9% purity) and ammonium hydroxide (NH₄OH, 25% in H₂O) were procured from Sigma-Aldrich. Nutrient agar and Mueller-Hinton agar/broth were obtained from HiMedia Laboratories for antibacterial tests. All chemicals were of analytical grade and used as received without further purification.

2.2. Synthesis of SnO₂ Nanoparticles

SnO₂ nanoparticles were synthesized by a facile chemical precipitation method at room temperature. In a typical procedure, 0.1 M aqueous solution of SnCl₂·2H₂O precursor was prepared by dissolving 1.89 g of tin(II) chloride

dihydrate in 100 mL of deionized water under constant stirring. Ammonium hydroxide was added dropwise to the salt solution until pH 10 to generate white precipitates of tin hydroxide. The precipitate was aged for 30 min under continuous stirring, filtered, and washed thoroughly with deionized water 3-4 times. The filtered precipitate was dried overnight at 100°C in a hot air oven to remove moisture. Finally, the dried precursor was calcined at 300°C, 400°C and 500°C for 1 h each in ambient air to obtain pure tetragonal SnO₂ nanoparticles.

2.3. Characterization

The crystallinity and phase purity of the as-synthesized and calcined SnO₂ nanoparticles were examined by X-ray diffraction (XRD, Shimadzu X6000 diffractometer) using CuK α radiation ($\lambda=1.5418$ Å). The XRD patterns were recorded at 40 kV and 30 mA over a 2θ range of 20°-80° at a scan rate of 2°/min. The average crystallite sizes were estimated using the Scherrer equation. The surface morphology was studied by scanning electron microscopy (SEM, VegaIII TESCAN). The chemical bonding and vibrational modes were analyzed by Fourier transform infrared spectroscopy (FTIR, Bruker Alpha II) over 400-4000 cm⁻¹ range at 4 cm⁻¹ resolutions.

2.4. Antibacterial Testing

The antibacterial activity of the calcined SnO₂ nanoparticles was evaluated against Gram-negative *Escherichia coli* (*E. coli*) and Gram-positive *Staphylococcus aureus* (*S. aureus*) by standard zone of inhibition method using Mueller-Hinton agar medium. Both bacterial strains were grown in Mueller-Hinton broth at 37°C for 17-19 h. The inhibition zone diameters were measured to determine the activity.

3. Results and Discussion

3.1. Structural Characterization

The phase evolution and crystallinity of the as-synthesized and calcined SnO₂ nanoparticles were investigated by powder X-ray diffraction (XRD) measurements using CuK α radiation ($\lambda = 1.5418$ Å). Prior to calcination, the XRD pattern of the precursor sample heated to 100°C showed the formation of an intermediate grayish beige colored hydro-tin oxide (Sn₃O₂(OH)₂) phase as the major component along with minor peaks corresponding to SnO and residual unreacted SnCl₂·2H₂O impurities (Figure 1). The broad overlapped peaks indicate the partially hydrolyzed nature of the as-prepared sample. The minor reflections at 2θ values of 33.3°, 50.7°, 62.5°, and 67.7° can be indexed to the (110), (112), (103) and (113) planes of tetragonal SnO (ICDD #06-0395). This indicates partial hydrolysis of the tin chloride precursor upon addition of ammonia to form the oxyhydroxide intermediate. The emergence of hydro-tin oxide (Sn₃O₂(OH)₂) as the predominant phase confirms partial hydrolysis of the SnCl₂·2H₂O precursor has occurred following ammonia addition, leading to formation of the oxyhydroxide intermediate. The minor presence of residual SnCl₂·2H₂O and SnO indicates the hydrolysis reaction is incomplete at 100°C. This demonstrates the as-prepared precursor consists of poorly crystalline Sn₃O₂(OH)₂ along with traces of unreacted starting materials. Subsequent calcination is necessary to obtain phase pure tetragonal SnO₂, as corroborated by the XRD patterns at higher temperatures. The observation of minor SnCl₂·2H₂O and SnO impurities provides insights into the reaction kinetics and hydrolysis pathway involved in the low temperature synthesis. Upon calcination from 300°C to 500°C, the XRD patterns confirmed successful phase transition to tetragonal rutile SnO₂ as the major phase (ICDD # 41-1445) along with significant improvement in crystallinity. No unidentified impurity peaks were observed suggesting high phase purity of the calcined SnO₂ nanoparticles. The prominent (110), (101) and (211) reflections correspond to the tetragonal SnO₂ phase. The average crystallite size (*D*) was estimated from the XRD peak broadening using the Scherrer equation [32]:

$$D = 0.9\lambda/\beta\cos\theta \quad (1)$$

where λ is the X-ray wavelength, β is the full width at half maximum (FWHM) of the diffraction peak, and θ is the Bragg angle.

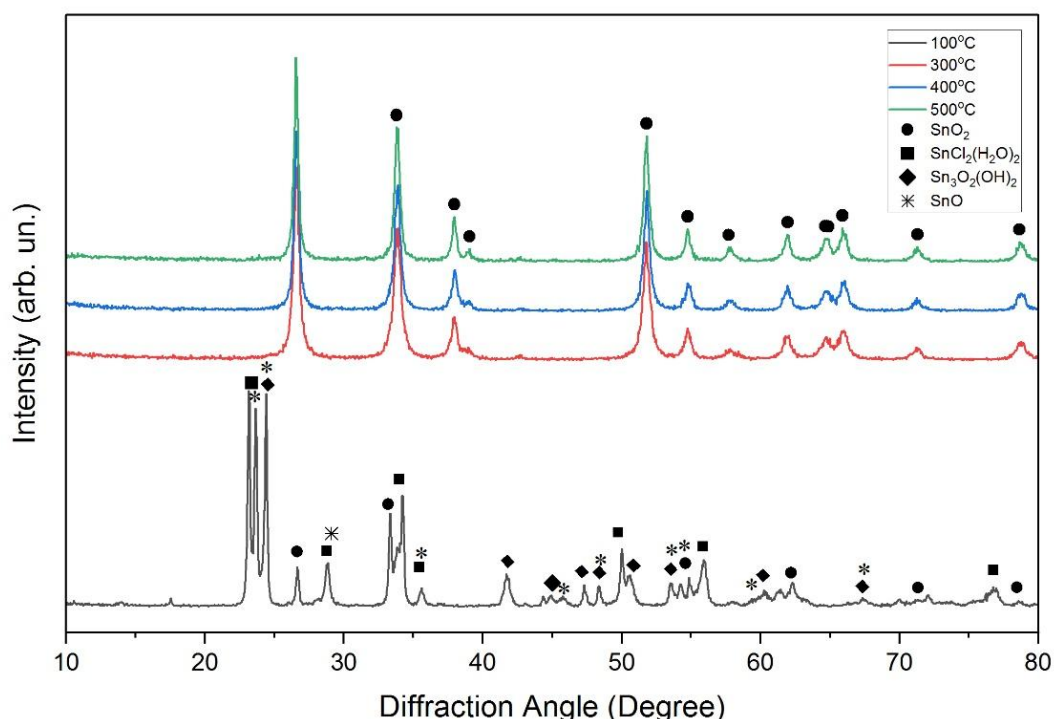


Figure 1: Evolution of SnO_2 phase purity with calcination temperature. XRD pattern of precursor at 100°C shows $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{Sn}_3\text{O}_2(\text{OH})_2$ and SnO phases. Calcined SnO_2 at 300°C , 400°C and 500°C exhibits tetragonal SnO_2 as dominant phase.

The peak breadth decreased progressively with increasing calcination temperature from 300°C to 500°C indicating crystallite growth. Correspondingly, the crystallite size increased from 11.9 nm to 13.9 nm and 17.2 nm for the samples calcined at 300°C , 400°C and 500°C respectively, as summarized in Table 1. The particle growth is likely due to agglomeration and sintering effects at higher temperatures. Additionally, the enhanced intensity and reduced FWHM of the XRD peaks points to improved crystallinity at higher calcination temperatures owing to increased ordering of the atomic planes and conversion of amorphous fractions to crystalline SnO_2 phase. The average lattice parameters calculated using the (hkl) plane spacing (d) were determined to be $a = b = 4.732 \text{ \AA}$ and $c = 3.660 \text{ \AA}$, consistent with tetragonal SnO_2 . The XRD analysis confirmed high purity tetragonal SnO_2 nanoparticles were obtained after thermal calcination with systematic crystallite growth at higher temperatures.

Table 1: Variation in crystallite size of SnO_2 nanoparticles with calcination temperature estimated from XRD data using Scherrer equation.

Calcination Temperature ($^\circ\text{C}$)	2Theta (Deg)	D (\AA)	FWHM (Deg)	(hkl)	Crystallite Size (nm)
300	26.608	3.347	0.489	110	13.01
	33.864	2.645	0.602	101	10.79
	51.765	1.765	0.575	211	12.01
400	26.615	3.347	0.437	110	13.03
	33.874	2.644	0.480	101	13.53
	51.774	1.764	0.467	211	14.78
500	26.593	3.349	0.348	110	18.35
	33.858	2.645	0.418	101	15.54
	51.764	1.765	0.389	211	17.76

The evolution of surface morphology and microstructure of the as-synthesized and thermally annealed SnO₂ nanopowder were thoroughly investigated using scanning electron microscopy (SEM). Significant changes in particle morphology and microstructure were observed in the SEM micrographs after calcination from 300°C to 500°C (Figure 2). At 300°C, partial fusion of nanoparticles into larger irregular clusters occurred leading to surface smoothing, indicative of initial sintering. The average particle size increased to ~68 nm. Further rise in calcination temperature to 400°C showed increased densification and grain growth, as evidenced by larger nanoparticles of ~87 nm size and distinct grain boundaries. Well-defined facets and polyhedral morphologies were observed in the 500°C calcined sample with average particle size of ~93 nm.

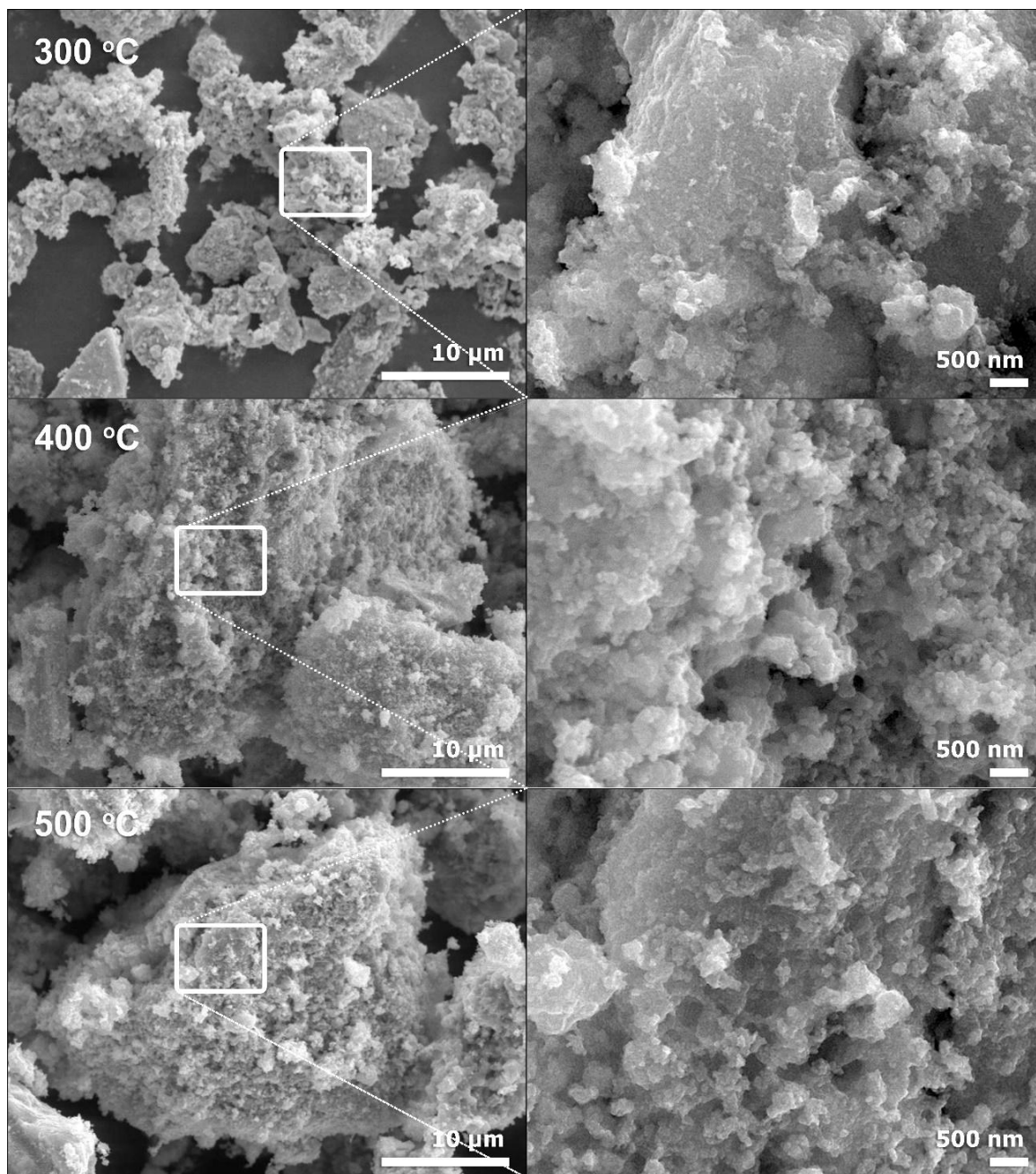


Figure 2: Microstructural evolution of SnO₂ nanoparticles with calcination temperature. SEM micrographs at low and high magnification showing morphology and agglomeration at 300°C, 400°C, and 500°C.

Image analysis was conducted using MountainsMap software by measuring over 100 particles from the SEM micrographs. This quantitative analysis reaffirmed the particle enlargement at higher calcination temperatures.

The sintering led to reduction in porosity and surface area. The particle growth likely occurs due to agglomeration, interparticle fusion, and Ostwald ripening effects which are accelerated at elevated temperatures [33]. The SEM findings corroborate well with the crystallite growth inferred from XRD peak narrowing. The smaller particle size and higher specific surface area of the 300°C sample would facilitate increased surface redox reactions. In contrast, the surface available for catalytic antibacterial activity may be restricted in the larger particles of the 400°C and 500°C samples. Thus, SEM provided critical insights into the surface morphology, microstructure evolution, and sintering dynamics of nanocrystalline SnO₂ particles with the variation in thermal processing conditions. The EDS spectra in Figure 3 confirm the presence of tin (Sn) and oxygen (O) as the predominant elements in all three SnO₂ samples calcined at 300°C, 400°C, and 500°C. This verifies the chemical composition aligns with pure SnO₂, as expected from the synthesis method. Additionally, residual traces of chlorine (Cl) originating from the tin chloride precursor are observed. The Cl content exhibits a declining trend from 0.9 wt% in the 300°C sample to 0.21 wt% in the 500°C sample. The reduction of Cl impurity with higher calcination temperature indicates more thorough removal of remnant precursor and improved sample purity. Estimation of the atomic Sn/O ratios from the EDS data reveals values approaching the ideal stoichiometric ratio of 0.5 for SnO₂ across all samples. The proximity of measured Sn/O ratios to 0.5 confirms the precipitation and calcination approach yields near-stoichiometric SnO₂ nanoparticles without substantial oxygen vacancies. In summary, the EDS elemental analysis verifies successful synthesis of stoichiometric SnO₂ nanopowder after calcination, with systematic decreases in residual Cl impurities at higher temperatures. The results validate the phase purity and composition of the SnO₂ nanoparticles obtained through the low-cost, eco-friendly chemical precipitation route.

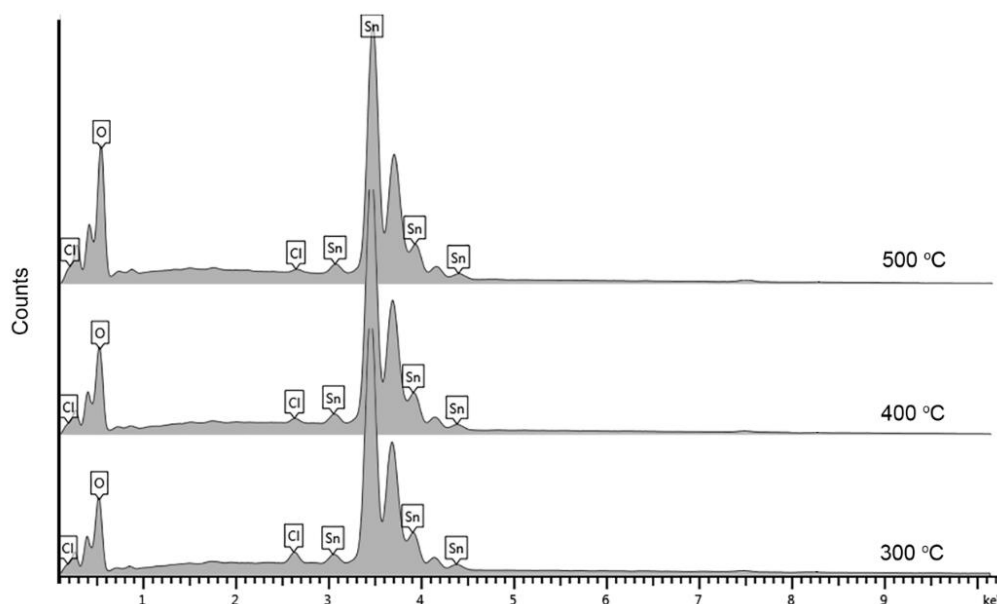


Figure 3: Elemental analysis of SnO₂ nanoparticles by EDS. Spectra for the samples calcined at 300°C, 400°C and 500°C confirm presence of Sn, O as major elements and trace Cl.

Fourier transform infrared (FTIR) spectroscopy elucidated the progression of chemical bonding configurations and vibrational fingerprints in the as-synthesized and calcined SnO₂ nanoparticles (Figure 4). The precursor sample heated to 100°C exhibited a broad envelope centered at 3348 cm⁻¹ attributed to the O-H stretching vibrations of absorbed water molecules and N-H stretching of ammonium residues from the synthesis [34, 35]. The peaks at 1606 cm⁻¹, 714 cm⁻¹ and 537 cm⁻¹ are ascribed to the H-O-H bending, Sn-O stretching, and Sn-Cl stretching modes respectively [36, 37]. The emergence of these bands corroborates the formation of a hydrated oxyhydroxide intermediate phase along with unreacted tin chloride precursor, as inferred from XRD patterns. Upon calcination to 300°C, the O-H and N-H peaks diminished indicating desorption of physisorbed species. Simultaneously, new asymmetric CO₂ stretches manifested at 2341 cm⁻¹ and 2360 cm⁻¹ due to atmospheric carbon dioxide. The broad envelope spanning 500-850 cm⁻¹ originates from overlapped metal-oxygen lattice vibrations of nanocrystalline SnO₂. The intense peak at 608 cm⁻¹ accompanied by a shoulder around 459 cm⁻¹

represents the asymmetric and symmetric O-Sn-O bridging stretches respectively [31, 35, 38]. The surface Sn-O-Sn bending mode contributes to the band at 855 cm^{-1} [36, 39]. With increasing calcination temperature to 400°C and 500°C , incremental blue-shifting and sharpening of these Sn-O-Sn and O-Sn-O peaks occurred, congruent with improved crystallinity and particle growth as evidenced in XRD and SEM. Concurrently, the Sn-Cl stretching mode at 537 cm^{-1} faded and disappeared by 300°C , indicative of complete hydrolysis of tin chloride to stoichiometric SnO_2 , further validated by declining chlorine content in EDS analysis. The FTIR analysis substantiated the chemical pathway involving thermal desorption of precursor species and crystallization of nanocrystalline SnO_2 exhibiting characteristic vibrational fingerprints. The trends align well with phase evolution observed using diffraction and microscopy techniques.

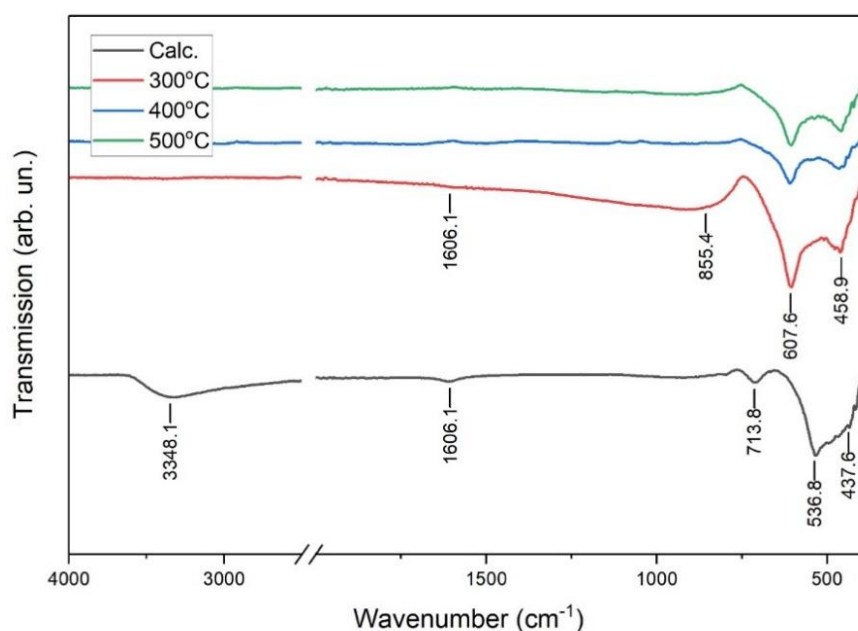


Figure 4. Calcination induced changes in FTIR spectra of SnO_2 . Broad envelope from $500\text{--}700\text{ cm}^{-1}$ represents Sn-O-Sn and O-Sn-O vibrational fingerprints in samples treated at 100°C , 300°C , 400°C and 500°C .

3.2. Antibacterial Performance

The antibacterial activity of the SnO_2 nanoparticles calcined at 300°C , 400°C and 500°C was evaluated against model Gram-negative *Escherichia coli* (*E. coli*) and Gram-positive *Staphylococcus aureus* (*S. aureus*) bacterial strains using standard agar well diffusion assays [40, 41]. As seen in the zone of inhibition (ZOI) results (Figure 5), the SnO_2 nanoparticles exhibited excellent antibacterial power against both *E. coli* and *S. aureus*, as evident from the clear ZOIs formed around the nanoparticle-loaded wells. The extent of ZOI gave quantitative indication of the antibacterial activity. Interestingly, an inverse correlation between the SnO_2 crystallite size and antibacterial activity was observed. The 300°C calcined sample with smallest crystallite size of 11.9 nm showed maximum inhibition zone diameters of 25 mm and 29 mm for *E. coli* and *S. aureus* respectively. The enhanced antibacterial effect of the lower crystallite size SnO_2 sample is attributed to the higher surface area and increased density of surface defects such as oxygen vacancies. This can promote generation of reactive oxygen species (ROS) like $\cdot\text{OH}$, $\text{O}_2^{\cdot-}$ and H_2O_2 when the particles are irradiated or upon interaction with bacterial cells. ROS can damage the cell membrane and intracellular components leading to leakage of cytoplasmic content and cell death. Additional mechanisms involve direct oxidative damage of cell wall/membrane by SnO_2 nanoparticle interaction as well as release of Sn^{2+} ions.

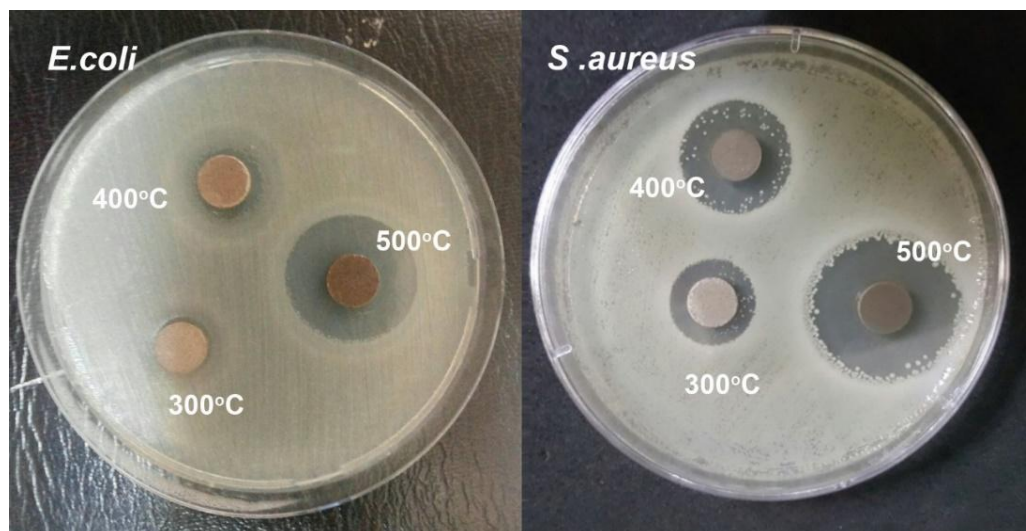


Figure 5: Photographs showing antibacterial activity of SnO₂ nanoparticles calcined at different temperatures against *E. coli* and *S. aureus* by zone of inhibition method.

Another factor is the cell wall structure of the bacterial strains. The thicker peptidoglycan layer of Gram-positive *S. aureus* could be more prone to ROS attack compared to the lipopolysaccharide membrane of Gram-negative *E. coli*. The results demonstrate that the SnO₂ nanoparticle crystallite size can be effectively tailored by tuning the calcination temperature to achieve optimal antibacterial activity against both Gram-types of pathogenic bacteria. The data in Table 2 shows the relationship between the crystallite size of calcined SnO₂ nanoparticles and their antibacterial activity against *E. coli* and *S. aureus*, as measured by inhibition zone diameter. As the calcination temperature was increased from 300°C to 500°C, the crystallite size of the SnO₂ nanoparticles systematically grew larger, from 11.9 nm to 17.2 nm, due to sintering and particle growth effects.

Table 2: Correlation between crystallite size of calcined SnO₂ nanoparticles and inhibition zone diameter for *E. coli* and *S. aureus* bacterial strains.

Calcinations Temperature °C	Crystallite Size (nm)	ZOI (mm) <i>E. coli</i>	ZOI (mm) <i>S. aureus</i>
300	11.93	25	29
400	13.87	13	22
500	17.21	11	17

Correspondingly, the inhibition zone diameters for both bacterial strains consistently decreased with increasing SnO₂ crystallite size. The sample calcined at 300°C, which had the smallest crystallite size of 11.9 nm, exhibited the largest inhibition zones of 25 mm for *E. coli* and 29 mm for *S. aureus*. In contrast, the sample calcined at 500°C, with the largest crystallite size of 17.2 nm, showed the narrowest inhibition zones of 11 mm for *E. coli* and 17 mm for *S. aureus*. This inverse relationship demonstrates that the antibacterial activity is reduced as the crystallite size of the SnO₂ nanoparticles increases. The enhanced antibacterial activity of smaller SnO₂ crystallites can be attributed to their larger surface area and higher density of surface defects, which can more effectively generate reactive oxygen species. Table 3 compiles the antibacterial activity of SnO₂ nanoparticles synthesized by various methods reported in literature against different microorganisms. It can be observed that the precipitation synthesized SnO₂ NPs in this work exhibit comparable or superior antibacterial activity relative to SnO₂ prepared by other more complex techniques. The SnO₂ NPs synthesized via precipitation in this study showed zone of inhibition ranging from 11-29 mm against *E. coli* and *S. aureus*. This activity is similar to SnO₂ NPs of comparable size prepared by co-precipitation (10-22 mm ZOI) [25, 26], precipitation (6-15 mm ZOI) [30], sol-gel (2-18 mm ZOI) [18] and other methods (8-24 mm ZOI) [27–29, 31]. The excellent antibacterial performance of the simply precipitated SnO₂ NPs demonstrates that high crystallinity and appropriate

nanoparticle size are more critical factors than synthesis technique. The precipitation method produces high purity crystalline SnO₂ NPs of suitable size to penetrate bacteria cells and generate ROS to damage cellular components. The table highlights the potential of these facile precipitation synthesized SnO₂ NPs as antibacterial agents against both Gram-positive and Gram-negative pathogenic bacteria.

Table 3: Comparison of antibacterial activity (zone of inhibition, ZOI) of SnO₂ nanoparticles.

Synthesis Method	Composition	Crystallite Size (nm)	Microorganism	ZOI (mm)	Ref.
Co-precipitation	SnO ₂ , CuO doped SnO ₂	10-15	E-coli K-neumo S-aureus	10	[25]
Co-precipitation	SnO ₂ , Co doped SnO ₂	24.8	E-coli B-subtilis	16-22	[26]
Precipitation	SnO ₂	NPs	E-coli S-aureus	6-15	[30]
Sol-Gel	SnO ₂	9-10	E-coli Micrococcus luteus	2-18	[18]
Precipitation	SnO ₂	18.79	E-coli B-subtilis Pseudomonas aeruginosa	8-12	[31]
Biosynthesis	SnO ₂	35	E-coli B-subtilis S-aureus Pseudomonas aeruginosa	16-24	[28]
Biosynthesis	SnO ₂	18.2	E-coli B-subtilis S-aureus S. pyogenes K. pneumoniae	9-15	[29]
Ultra-sonochemical	SnO ₂	5-30	E-coli C. albicans	14-22	[27]
Precipitation	SnO ₂	12-17	E-coli S-aureus	11-29	Current work

4. Conclusions

In this work, SnO₂ nanoparticles were successfully synthesized via a simple chemical precipitation route using tin chloride and ammonia precursors at room temperature. XRD studies confirmed the formation of tetragonal SnO₂ with high phase purity after calcination from 300°C to 500°C. The average crystallite size systematically increased from 11.9 nm to 17.2 nm with higher calcination temperature as a result of sintering. FTIR spectra revealed the characteristic Sn-O and O-Sn-O vibrational modes in the samples. SEM micrographs demonstrated the particle size enlargement from ~68 nm to ~93 nm upon calcining from 300°C to 500°C due to agglomeration and Ostwald ripening effects. The antibacterial activity of the calcined SnO₂ nanoparticles against *E. coli* and *S. aureus* was found to be inversely related to the crystallite size. The sample calcined at lower temperature of 300°C with smallest crystallite size of 11.9 nm exhibited maximum inhibition zones of 25 mm and 29 mm against *E. coli* and *S. aureus* respectively. The higher surface area and surface defects in smaller crystallites can promote reactive oxygen species generation, thereby enhancing the antibacterial behaviour. This work demonstrates that the SnO₂ nanoparticle crystallinity and morphology can be effectively tailored by tuning the calcination temperature to achieve optimal antibacterial action against both Gram-positive and Gram-negative pathogenic bacteria. The green chemistry approach presents a facile route for controlling the antibacterial activity of SnO₂ nanomaterials for water treatment and food packaging applications.

Acknowledgement

The authors thank the Center of Advanced Materials at the Materials Research Directorate for their support of this work. The facilities and resources provided have been invaluable, allowing us to synthesize and characterize the SnO₂ nanoparticles.

Conflict of Interest

The authors declare that they have no conflict of interest.

References

- [1] Das, S., and Jayaraman, V. "SnO₂: A Comprehensive Review on Structures and Gas Sensors." *Progress in Materials Science*, vol. 66, 2014, pp. 112–255. <https://doi.org/10.1016/j.pmatsci.2014.06.003>.
- [2] Ismail, R. A., Erten-Ela, S., Ali, A. K., Yavuz, C., and Hassoon, K. I. "Pulsed Laser Ablation of Tin Oxide Nanoparticles in Liquid for Optoelectronic Devices." *Silicon*, vol. 13, no. 9, 2021, pp. 3229–3237. <https://doi.org/10.1007/s12633-020-00676-y>.
- [3] Bhawna, Choudhary, A. K., Gupta, A., Kumar, S., Kumar, P., Singh, R. P., Singh, P., and Kumar, V. "Synthesis, Antimicrobial Activity, and Photocatalytic Performance of Ce Doped SnO₂ Nanoparticles." *Frontiers in Nanotechnology*, vol. 2, no. 2, 2020, pp. 1–7. <https://doi.org/10.3389/fnano.2020.595352>.
- [4] Kamble, D. L., Harale, N. S., Patil, V. L., Patil, P. S., and Kadam, L. D. "Characterization and NO₂ Gas Sensing Properties of Spray Pyrolyzed SnO₂ Thin Films." *Journal of Analytical and Applied Pyrolysis*, vol. 127, no. 2, 2017, pp. 38–46. <https://doi.org/10.1016/j.jaap.2017.09.004>.
- [5] Zarkov, A., Stanulis, A., Mikoliunaite, L., Salak, A. N., and Kareiva, A. "Organic-Free Synthesis of Nanostructured SnO₂ Thin Films by Chemical Solution Deposition." *Thin Solid Films*, vol. 649, 2018, pp. 219–224. <https://doi.org/10.1016/j.tsf.2018.01.056>.
- [6] Abdelkrim, A., Rahmane, S., Abdelouahab, O., Abdelmalek, N., and Brahim, G. "Effect of Solution Concentration on the Structural, Optical and Electrical Properties of SnO₂ Thin Films Prepared by Spray Pyrolysis." *Optik*, vol. 127, no. 5, 2016, pp. 2653–2658. <https://doi.org/10.1016/j.ijleo.2015.11.232>.
- [7] Abdulsattar, M. A., Batros, S. S., and Addie, A. J. "Indium Doped SnO₂ nanostructures Preparation and Properties Supported by DFT Study." *Superlattices and Microstructures*, vol. 100, 2016, pp. 342–349. <https://doi.org/10.1016/j.spmi.2016.09.042>.
- [8] Hassan, A. I., Addie, A. J., and Admon, J. "Influence of Precursor Concentration on the Optoelectronic Properties of Spray Deposited SnO₂/Si Heterojunction." *Materials Research Express*, vol. 6, no. 9, 2019, p. 0950c4. <https://doi.org/10.1088/2053-1591/ab350b>.
- [9] Chowdhury, F. R., Choudhury, S., Hasan, F., and Begum, T. "Optical Properties of Undoped and Indium-Doped Tin Oxide Thin Films." *Journal of Bangladesh Academy of Sciences*, vol. 35, no. 1, 2013, pp. 99–111. <https://doi.org/10.3329/jbas.v35i1.7975>.
- [10] Ji, Z., He, Z., Song, Y., Liu, K., and Ye, Z. "Fabrication and Characterization of Indium-Doped p-Type SnO₂ Thin Films." *Journal of Crystal Growth*, vol. 259, no. 3, 2003, pp. 282–285. <https://doi.org/10.1016/j.jcrysgro.2003.07.003>.
- [11] Li, Z., Shen, W., Zhang, X., Fang, L., and Zu, X. "Controllable Growth of SnO₂ Nanoparticles by Citric Acid Assisted Hydrothermal Process." *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, vol. 327, Nos. 1–3, 2008, pp. 17–20. <https://doi.org/10.1016/j.colsurfa.2008.05.043>.

- [12] Drzymala, E., Gruzel, G., Depciuch, J., Budziak, A., Kowal, A., and Parlinska-Wojtan, M. "Structural, Chemical and Optical Properties of SnO₂ NPs Obtained by Three Different Synthesis Routes." *Journal of Physics and Chemistry of Solids*, vol. 107, 2017, pp. 100–107. <https://doi.org/10.1016/j.jpcs.2017.03.026>.
- [13] T. Lamdhade, G., C. Raghuwanshi, F., M. Agrawal, R., M. Balkhande, V., and Shripath, T. "SnO₂ Nanoparticles Synthesis Via Liquid-Phase Co-Precipitation Technique." *Advanced Materials Letters*, vol. 6, no. 8, 2015, pp. 738–742. <https://doi.org/10.5185/amlett.2015.5877>.
- [14] Chen, W., Ghosh, D., and Chen, S. "Large-Scale Electrochemical Synthesis of SnO₂ Nanoparticles." *Journal of Materials Science*, vol. 43, no. 15, 2008, pp. 5291–5299. <https://doi.org/10.1007/s10853-008-2792-x>.
- [15] Ravichandran, K., Shalini, R., Ayyanar, M., Kavitha, P., Baneto, M., Karunakaran, M., Praseetha, P. K., Pushpa, K. C. S., and Anuradha, N. "Effect of PH of the Precursor Solution on the Photocatalytic and Biomedical Applications of Enzyme Coupled ZnO and SnO₂ Nanomaterials: A Comparative Study." *Journal of Water Process Engineering*, vol. 53, no. May, 2023, p. 103817. <https://doi.org/10.1016/j.jwpe.2023.103817>.
- [16] Zhang, Q., Zou, X., Wang, Y., and Habibi, M. "Study on Photocatalytic, Electric, and Sensing Behavior of Co- and Ag-Codoped Tin Dioxide (SnO₂) Nano Particles." *Materials Science and Engineering: B*, vol. 296, no. April, 2023, p. 116687. <https://doi.org/10.1016/j.mseb.2023.116687>.
- [17] Batzill, M., and Diebold, U. "The Surface and Materials Science of Tin Oxide." *Progress in Surface Science*, vol. 79, Nos. 2–4, 2005, pp. 47–154. <https://doi.org/10.1016/j.progsurf.2005.09.002>.
- [18] Bilal Ahmad, T., Asif, A. B., Atif Khurshid, W., Masood, A. K., and Gulzar Ahmad, S. "Preparation and Characterization of SnO₂ Nanoparticles for Antibacterial Properties." *Nanomaterial Chemistry and Technology*, vol. 2, no. 1, 2020, pp. 1–5. <https://doi.org/10.33805/2690-2575.109>.
- [19] Yuliarto, B., Gumilar, G., Zuhendri, D. W., Nugraha, and Septiani, N. L. W. "Preparation of SnO₂ Thin Film Nanostructure for CO Gas Sensor Using Ultrasonic Spray Pyrolysis and Chemical Bath Deposition Technique." *Acta Physica Polonica A*, vol. 131, no. 3, 2017, pp. 534–538. <https://doi.org/10.12693/APhysPolA.131.534>.
- [20] Galal, A., Hassan, H. K., Atta, N. F., Abdel-Mageed, A. M., and Jacob, T. "Synthesis, Structural and Morphological Characterizations of Nano-Ru-Based Perovskites/RGO Composites." *Scientific Reports*, vol. 9, no. 1, 2019, pp. 1–13. <https://doi.org/10.1038/s41598-019-43726-1>.
- [21] Meena Kumari, M., and Philip, D. "Synthesis of Biogenic SnO₂ Nanoparticles and Evaluation of Thermal, Rheological, Antibacterial and Antioxidant Activities." *Powder Technology*, vol. 270, 2015, pp. 312–319. <https://doi.org/10.1016/j.powtec.2014.10.034>.
- [22] Samrot, A. V., Sahithya, C. S., Selvarani A, J., Purayil, S. K., and Ponnaiah, P. "A Review on Synthesis, Characterization and Potential Biological Applications of Superparamagnetic Iron Oxide Nanoparticles." *Current Research in Green and Sustainable Chemistry*, vol. 4, no. December 2020, 2021, p. 100042. <https://doi.org/10.1016/j.crgsc.2020.100042>.
- [23] Amininezhad, S. M., Rezvani, A., Amouheidari, M., Amininejad, S. M., and Rakhshani, S. "The Antibacterial Activity of SnO₂ Nanoparticles against Escherichia Coli and Staphylococcus Aureus." *Zahedan Journal of Research in Medical Sciences*, vol. 17, no. 9, 2015. <https://doi.org/10.17795/zjrms-1053>.

- [24] Cavaliere, E., De Cesari, S., Landini, G., Riccobono, E., Pallecchi, L., Rossolini, G. M., and Gavioli, L. "Highly Bactericidal Ag Nanoparticle Films Obtained by Cluster Beam Deposition." *Nanomedicine: Nanotechnology, Biology, and Medicine*, vol. 11, no. 6, 2015, pp. 1417–1423. <https://doi.org/10.1016/j.nano.2015.02.023>.
- [25] Gopal, S., Iruson, B., Balaraman, S., Krishnmoorthy, S., and Elayaperumal, M. "Effect on Optical and Antibacterial Activity of SnO₂ and CuO Blended SnO₂ Nanoparticles." *Soft Nanoscience Letters*, vol. 13, no. 02, 2023, pp. 1–12. <https://doi.org/10.4236/snl.2023.132001>.
- [26] Qamar, M., Shahid, S., Khan, S. A., Zaman, S., and Sarwar, M. N. "SYNTHESIS CHARACTERIZATION , OPTICAL AND ANTIBACTERIAL STUDIES OF Co-DOPED SnO₂ NANOPARTICLES." *Digest Journal of Nanomaterials and Biostructures*, vol. 12, no. 4, 2017, pp. 1127–1135.
- [27] Rehman, S., Asiri, S. M., Khan, F. A., Jermy, B. R., Khan, H., Akhtar, S., Jindan, R. Al, Khan, K. M., and Qurashi, A. "Biocompatible Tin Oxide Nanoparticles: Synthesis, Antibacterial, Anticandidal and Cytotoxic Activities." *ChemistrySelect*, vol. 4, no. 14, 2019, pp. 4013–4017. <https://doi.org/10.1002/slct.201803550>.
- [28] Al-Enazi, N. M., Ameen, F., Alsamhary, K., Dawoud, T., Al-Khattaf, F., and AlNadhari, S. "Tin Oxide Nanoparticles (SnO₂-NPs) Synthesis Using Galaxaura Elongata and Its Anti-Microbial and Cytotoxicity Study: A Greenery Approach." *Applied Nanoscience (Switzerland)*, vol. 13, no. 1, 2023, pp. 519–527. <https://doi.org/10.1007/s13204-021-01828-1>.
- [29] Fatimah, I., Purwiandono, G., Hidayat, H., Sagadevan, S., Ghazali, S. A. I. S. M., Oh, W. C., and Doong, R. A. "Flower-like SnO₂ Nanoparticle Biofabrication Using Pometia Pinnata Leaf Extract and Study on Its Photocatalytic and Antibacterial Activities." *Nanomaterials*, vol. 11, no. 11, 2021. <https://doi.org/10.3390/nano11113012>.
- [30] Khanoma, R., Parveenb, S., and Hasan, M. "Antimicrobial Activity of SnO₂ Nanoparticles against Escherichia Coli and Staphylococcus Aureus and Conventional Antibiotics." *American Scientific Research Journal for Engineering, Technology, and Sciences*, vol. 46, no. 1, 2018, pp. 111–121.
- [31] Abirami, S., Viruthagiri, G., and Ashokkumar, K. "Structural, Morphological and Anti - Bacterial Activities of Pure SnO₂ Nanoparticles Prepared by Chemical Precipitation Method." *Materials Today: Proceedings*, Vol. 73, 2023, pp. 535–538. <https://doi.org/10.1016/j.matpr.2022.11.442>.
- [32] Abdulsattar, M. A., Batros, S. S., and Addie, A. J. "Spectroscopic Properties of Indium-Doped CdO Nanostructures Supported by DFT Calculations." *Surface Review and Letters*, vol. 26, no. 04, 2019, p. 1850169. <https://doi.org/10.1142/S0218625X1850169X>.
- [33] Palasantzas, G., Vystavel, T., Koch, S. A., and De Hosson, J. T. M. "Coalescence Aspects of Cobalt Nanoparticles during in Situ High-Temperature Annealing." *Journal of Applied Physics*, vol. 99, no. 2, 2006. <https://doi.org/10.1063/1.2163983>.
- [34] Orel, B., Lavrenčič-Štankar, U., Crnjak-Orel, Z., Bukovec, P., and Kosec, M. "Structural and FTIR Spectroscopic Studies of Gel-Xerogel-Oxide Transitions of SnO₂ and SnO₂ : Sb Powders and Dip-Coated Films Prepared via Inorganic Sol-Gel Route." *Journal of Non-Crystalline Solids*, vol. 167, no. 3, 1994, pp. 272–288. [https://doi.org/10.1016/0022-3093\(94\)90250-X](https://doi.org/10.1016/0022-3093(94)90250-X).
- [35] Mahmood, H., Khan, M. A., Mohuddin, B., and Iqbal, T. "Solution-Phase Growth of Tin Oxide (SnO₂) Nanostructures: Structural, Optical and Photocatalytic Properties." *Materials Science and Engineering B: Solid-State Materials for Advanced Technology*, vol. 258, no. August 2018, 2020, p. 114568. <https://doi.org/10.1016/j.mseb.2020.114568>.

- [36] Sukriti, and Chand, P. “Effect of PH Values on the Structural, Optical and Electrical Properties of SnO₂ Nanostructures.” *Optik*, vol. 181, no. September 2018, 2019, pp. 768–778. <https://doi.org/10.1016/j.ijleo.2018.10.203>.
- [37] Keny, S. J., Srivastava, A. P., Debnath, A. K., Adhikari, S., and Rath, M. C. “One-Step Synthesis of Tin Oxide Nanoparticles in Aqueous Solution Induced by Free Radicals.” *Materials Chemistry and Physics*, vol. 286, o. December 2021, 2022, p. 126184. <https://doi.org/10.1016/j.matchemphys.2022.126184>.
- [38] Perumal, V., Inmozhi, C., Uthrakumar, R., Robert, R., Chandrasekar, M., Mohamed, S. B., Honey, S., Raja, A., Al-Mekhlafi, F. A., and Kaviyarasu, K. “Enhancing the Photocatalytic Performance of Surface - Treated SnO₂ Hierarchical Nanorods against Methylene Blue Dye under Solar Irradiation and Biological Degradation.” *Environmental Research*, vol. 209, no. September 2021, 2022, p. 112821. <https://doi.org/10.1016/j.envres.2022.112821>.
- [39] Yakout, S. M. “Engineering of Visible Light Photocatalytic Activity in SnO₂ Nanoparticles: Cu²⁺-Integrated Li⁺, Y³⁺ or Zr⁴⁺ Dopants.” *Optical Materials*, vol. 116, no. April, 2021, p. 111077. <https://doi.org/10.1016/j.optmat.2021.111077>.
- [40] Hikmet, R., and Hussein, N. “The Biological Activity of Mycosynthesized Silver Nanoparticles Against Some Pathogenic Bacteria.” *Journal of Applied Sciences and Nanotechnology*, vol. 2, no. 1, 2022, pp. 59–68. <https://doi.org/10.53293/jasn.2021.3963.1063>.
- [41] Khedaer, Z., Ahmed, D., and Al-Jawad, S. “Investigation of Morphological, Optical, and Antibacterial Properties of Hybrid ZnO-MWCNT Prepared by Sol-Gel.” *Journal of Applied Sciences and Nanotechnology*, vol. 1, no. 2, 2021, pp. 66–77. <https://doi.org/10.53293/jasn.2021.11634>.