



Photocatalytic degradation of methyl orange using biogenic Ag–ZnO nanostructures derived from *Searsia lancea*

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Abstract

The uncontrolled discharge of synthetic dyes such as methyl orange (MO) into aquatic systems poses significant environmental and health challenges due to their persistence and resistance to conventional treatment methods. In this study, Ag-doped ZnO nanoparticles were synthesized via a green and sustainable route using *Searsia lancea* leaf extract, with Ag doping levels of 1, 5, 10, and 20%. The materials were characterized for their structural, morphological, and optical properties. A successful incorporation of Ag into the ZnO lattice was confirmed by X-ray diffraction (XRD), Fourier transform infrared (FTIR), and Ultraviolet-visible spectroscopy (UV-vis spectroscopy). Ag doping induced a surface plasmon resonance effect, reducing the bandgap and extending light absorption from the UV to the visible region. The bandgap energy decreased from 3.36 eV (pristine) to 3.29, 3.00, and 2.76 eV with 1, 5, and 10% Ag doping, respectively, indicating modified electronic properties. The photocatalytic activity of the green-synthesised ZnO and Ag-doped ZnO catalysts was evaluated for MO degradation under visible light irradiation over 120 min. Pristine ZnO achieved 61.35% degradation, while Ag/ZnO exhibited significantly enhanced performance, with 1, 5, 10, and 20% doping achieving 93.20, 98.06, 99.35, and 95.85% efficiencies, respectively. The optimal 10% Ag/ZnO catalyst was further tested for the degradation of other pollutants, including crystal violet and ibuprofen, yielding efficiencies of 84.9% and 83.3%. The results highlight green synthesis as a viable route for producing highly active Ag-doped ZnO photocatalysts with application in wastewater treatment.

Keywords Ag-doping, ZnO nanoparticles · Green synthesis · *Searsia lancea* extract · Photocatalysis · Methyl orange

1 Introduction

The global water crisis remains one of the persistent challenges of our time, with increasing industrial pollution exacerbating the situation [1]. Direct water contamination from

industries, particularly textile, leather, paper, paint, plastic, food, and pharmaceutical sectors, has led to the widespread release of harmful residual chemicals such as dyes [2, 3]. Recent studies reported that around 2% of the dye produced during manufacturing is released into the effluent. Furthermore, industries that use these dyes lose about 15% of the dyes during the dyeing process [4, 5]. With many industries lacking adequate systems for primary dye removal, these effluents are often discharged into the environment [6]. This leads to significant contamination of water sources and poses serious risks to both aquatic ecosystems and human health [7]. Among these pollutants, azo dyes like methyl orange are particularly problematic due to their high solubility, persistence and resistance to conventional wastewater treatment methods, making their degradation a critical environmental challenge [8]. These synthetic dyes, characterized by their recalcitrant molecular structures, are not readily biodegradable. Consequently, they may remain in

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the environment, causing prolonged ecological harm and posing potential genotoxic risks [9]. The alarming rise in toxic industrial effluent underscores the urgent need for effective and sustainable wastewater treatment methods to ensure the continued provision of clean water, support the sustainable operations of industries and preserve the health of the ecosystem in line with achieving the respective Sustainable Development Goals.

Various methods have been employed to eliminate organic dyes from wastewater. These methods are classified into physical (adsorption, coagulation/flocculation, and membrane technology), chemical (photocatalysis, ozonation, and electro-Fenton processes), and biological (enzymatic degradation, microbial treatment, biosorption, and bioremediation) [10, 11]. Each technique has its own strengths and limitations that influence its practical application. However, many studies have shown that their large-scale use, especially in developing countries, remains limited due to inherent drawbacks. For example, physical methods mainly transfer dyes between phases without fully degrading them [12]. Chemical methods such as photocatalysis can effectively mineralize dyes but are often costly [13]. In contrast to biological methods, which are cost-effective and environmentally friendly, they tend to be slower [14]. Among the available methods, advanced oxidation processes (AOPs) have gained recognition as an effective and environmentally friendly approach for large-scale industrial water and wastewater treatment. These processes generate highly reactive hydroxyl radicals ($\bullet\text{OH}$), capable of degrading hazardous organic compounds into harmless by-products such as carbon dioxide and water. Among the various AOPs, semiconductor-based photocatalysis has gained considerable attention due to its simplicity, cost-effectiveness, and ability to achieve complete mineralization of organic dyes [15, 16]. Transition metal oxides, particularly zinc oxide nanoparticles (ZnO NPs), are recognized as a prototypical semiconductor that can be utilized in photocatalysis due to their unique physicochemical properties [17, 18]. Despite its promising photocatalytic potential, the utilization of ZnO nanoparticles still faces several challenges that hinder large-scale applications. These include (1) limited solar spectrum utilization due to wide band gap energy, (2) high electron recombination rates, and (3) extensive use of organic solvents in addition to energy-intensive synthesis procedures. Hence, resulting in restricted visible light absorption, reduced photocatalytic efficiency and high production cost, respectively [19, 20].

To address these challenges, different strategies such as the green synthesis approach and doping have been explored as effective strategies [21]. The electronic properties of ZnO are improved through doping with noble metals or other semiconducting elements, allowing for n-type or p-type

modifications [22]. Metal ion doping enhances ZnO's catalytic, magnetic, optical, thermal, and electrical properties by influencing its nanoparticle size, crystal structure, and surface-to-volume ratio [23–25]. Silver (Ag) is particularly considered an excellent dopant due to its ability to induce surface plasmon resonance (SPR) [25] and enhance carrier separation at the Schottky interface, which improves its photocatalytic performance [26]. Ag doping in ZnO is known to introduce oxygen vacancies, induce crystalline modifications, and affect light scattering behaviour.

Various physicochemical methods, such as sol-gel synthesis, oxidative reactions, thermal evaporation, co-precipitation, chemical vapour deposition, and sonochemical techniques, have been widely used to fabricate silver-doped zinc oxide (Ag-doped ZnO) nanoparticles [27–29]. However, these methods often involve toxic reagents or complex operational setups [30]. Green chemistry offers an eco-friendly alternative through biogenic synthesis, utilizing inherent phytochemicals such as flavonoids and tannins to act as both reducing and capping agents [31–33]. This eco-friendly approach aligns with green chemistry principles by utilizing renewable resources to operate under mild conditions, thereby avoiding toxic reagents, lowering energy consumption, and improving nanoparticle biocompatibility for biomedical use. Moreover, it is both cost-effective and scalable [34, 35]. Numerous plants, such as *Moringa oleifera*, *Psathyrella candolleana*, *Eucalyptus globulus*, and *Euphorbia stricta* L., have been successfully employed for the synthesis of ZnO nanoparticles [31, 32, 36, 37].

Despite the progress in green nanotechnology, there is a notable gap in the use of indigenous medicinal plants as bioresources for nanoparticle synthesis. *Searsia lancea* (*S. lancea*), commonly known as the Karee and belonging to the *Anacardiaceae* family (the Mango family), is rich in antioxidants and also possesses antibacterial properties [35, 38]. The plant is valued for its rapid growth, adaptability, and phytochemical richness, yet its potential for nanoparticle synthesis remains unexplored. This study addresses this gap by pioneering the use of *S. lancea* leaf extract as a green reducing and stabilizing agent for the fabrication of Ag nanoparticles, ZnO nanoparticles, and Ag/ZnO nanocomposites.

Consequently, this study reports the green synthesis of Ag/ZnO nanocomposites via *S. lancea* leaf extract and its photocatalytic activity for methyl orange degradation under visible light. The nanocomposite's structural, optical, and morphological properties were evaluated through various characterization techniques. Additionally, the study investigates how metal-ion doping influences the photocatalytic degradation efficiency. By leveraging the unique phytochemistry of *S. lancea*, this work establishes a sustainable method for synthesizing biocompatible nanomaterials with

improved bioactivity and stability. It also reveals a synergistic enhancement of photocatalytic activity in the Ag/ZnO nanocomposites, providing a green alternative to conventional chemical synthesis and highlighting their potential for sustainable wastewater treatment.

2 Methodology

2.1 Materials

The reagents used, including sodium hydroxide (NaOH), zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$), silver nitrate (AgNO_3), methyl orange (MO), and ethanol ($\text{C}_2\text{H}_5\text{OH}$), were obtained from Merck, South Africa. All chemicals and solvents used were of analytical grade and utilised without further purification. Fresh *Searsia lancea* leaves were obtained from Mmabatho, South Africa, and were properly identified. De-ionized water was employed in all synthesis reactions.

2.2 Preparation of *Searsia lancea* leaves extract

Fresh leaves of *Searsia lancea* were collected and sequentially rinsed with tap water, followed by distilled water to remove dust particles and surface impurities. The leaves were, thereafter, air-dried under shade at room temperature for three weeks until they were completely moisture-free. The dried leaves were cut into small pieces and finely ground using a blender. The plant extract was prepared by adding 15 g of the powdered leaves to 300 mL of deionised water in a 500 mL beaker. The mixture was subjected to steady heating and continuous stirring with a magnetic stirrer for 1 h at 80 °C to facilitate the release of phytochemicals into the solution. After cooling the mixture to room temperature, it was first sieved to remove residues and subsequently filtered to obtain a clear, particle-free extract. The cooled mixture was sieved and then filtered through filter paper to obtain a clear extract. This process minimized microbial contamination and safeguarded the bioactive compounds from degradation before their use in the synthesis process.

2.3 Synthesis of ZnO nanoparticles

ZnO nanoparticles were synthesised following a previously reported procedure [32], with some modifications. Firstly, a solution of 2.51 M of ($\text{Zn}(\text{CH}_3\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$) was mixed with 100 mL of plant extracts. NaOH was added to the resultant mixture to adjust the pH to 10, followed by stirring at 80 °C for 2 h in a beaker. Thereafter, it was centrifuged for 15 min at 5500 rpm, and the supernatant was removed. The obtained product was rinsed several times with distilled

water and once with ethanol, followed by drying overnight. The dried sample was then calcinated at 450 and 550 °C for 2 h to form an off-white product.

2.4 Synthesis of Ag-doped ZnO nanocomposite (Ag/ZnO)

Ag-doped ZnO NPs were synthesised following the procedure reported by Sharma et al. [39] with some modifications. In this method, 70 mL of 0.1 M zinc acetate solution ($\text{Zn}(\text{CH}_3\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$) was prepared, followed by the gradual addition of 10 mL of 0.01 M AgNO_3 and 20 mL of aqueous *S. lancea* leaf extract. Plant extract served as a reducing and stabilizing agent. Polyethylene glycol (PEG, 4%) was added dropwise under continuous stirring to serve as a capping agent. The mixture was maintained under magnetic stirring for 30 min, followed by an additional 30 min of stirring at 70 °C. The pH of the reaction medium was adjusted to 10 by the dropwise addition of 0.1 M NaOH, and stirring was maintained for 2 h at 70 °C. The resulting suspension was centrifuged at 5500 rpm for 20 min, and the precipitate obtained was washed several times with deionised water. The washed precipitate was dried in a hot-air oven at 100 °C for 2 h and subsequently calcined in a muffle furnace at 450 °C and 550 °C for 2 h. The synthesized sample was then used for further research applications.

2.5 Characterisation

The crystal structures of the synthesized ZnO NPs and Ag-doped ZnO nanocomposites were studied using a Bruker D8 Advance powder X-ray diffractometer, which employs Cu K α radiation ($\lambda = 1.5405 \text{ \AA}$) as the X-ray source and operates at a scanning rate of 1°/min. The crystallite sizes and lattice parameters, including unit dislocation density, cell volume, strain, and porosity, were calculated from the XRD data using X'Pert HighScore Plus. The morphological characterization of the nanomaterials was examined using a Zeiss Ultra Plus field emission scanning electron microscope (FE-SEM). Optical properties were studied using a PerkinElmer LAMBDA 1050+UV/Vis/NIR spectrophotometer. Functional groups of the synthesized nanomaterials were identified using Fourier-transform infrared spectroscopy (Bruker alpha-P FTIR spectrophotometer). These techniques provided comprehensive information on the morphological properties, optical behaviour, crystal structure and chemical composition of the synthesized nanomaterials.

2.6 Photocatalytic oxidation experiments

Catalytic oxidation experiments were conducted in a photocatalytic reactor system. In a typical procedure, 50 mL

of 10 mg/L dye solution was added to the reactor chamber together with a specific amount of the nanocatalyst. The mixture was stirred in the dark for 30 min to attain adsorption–desorption equilibrium and then exposed to a visible light source. During the photocatalytic reaction, sample aliquots were taken every 20 min, filtered to separate the catalyst from the dye solution and the absorbance was measured at 464 nm using a UV–vis spectrophotometer. The percentage removal of MO was calculated using Eq. (1). The kinetics study was carried out by fitting the degradation data to the linearized form of the pseudo-1st-order rate equation given in Eq. (2).

$$\% \text{Degradation} = \left(1 - \frac{A_t}{A_0}\right) \times 100 \quad (1)$$

Where A_0 and A_t are the initial and final absorbance of the solution at time t , respectively.

$$\ln C_t = \ln C_0 - kt \quad (2)$$

Where C_0 is the initial and C_t is the final conc. of the MO solution, k represents the apparent rate constant, and t represents the reaction time.

3 Results and discussion

3.1 Structural properties

The crystal structure, phase composition, and phase purity of the green-synthesized ZnO nanoparticles were analyzed using the XRD technique, and the results are presented in Figs. 1 (a and b). The diffraction patterns at the two different calcination temperatures revealed nine distinct peaks, all characteristic of the hexagonal ZnO with (JCPDS card no. 36–1451). These peaks appear at 2θ values of 31.71° ,

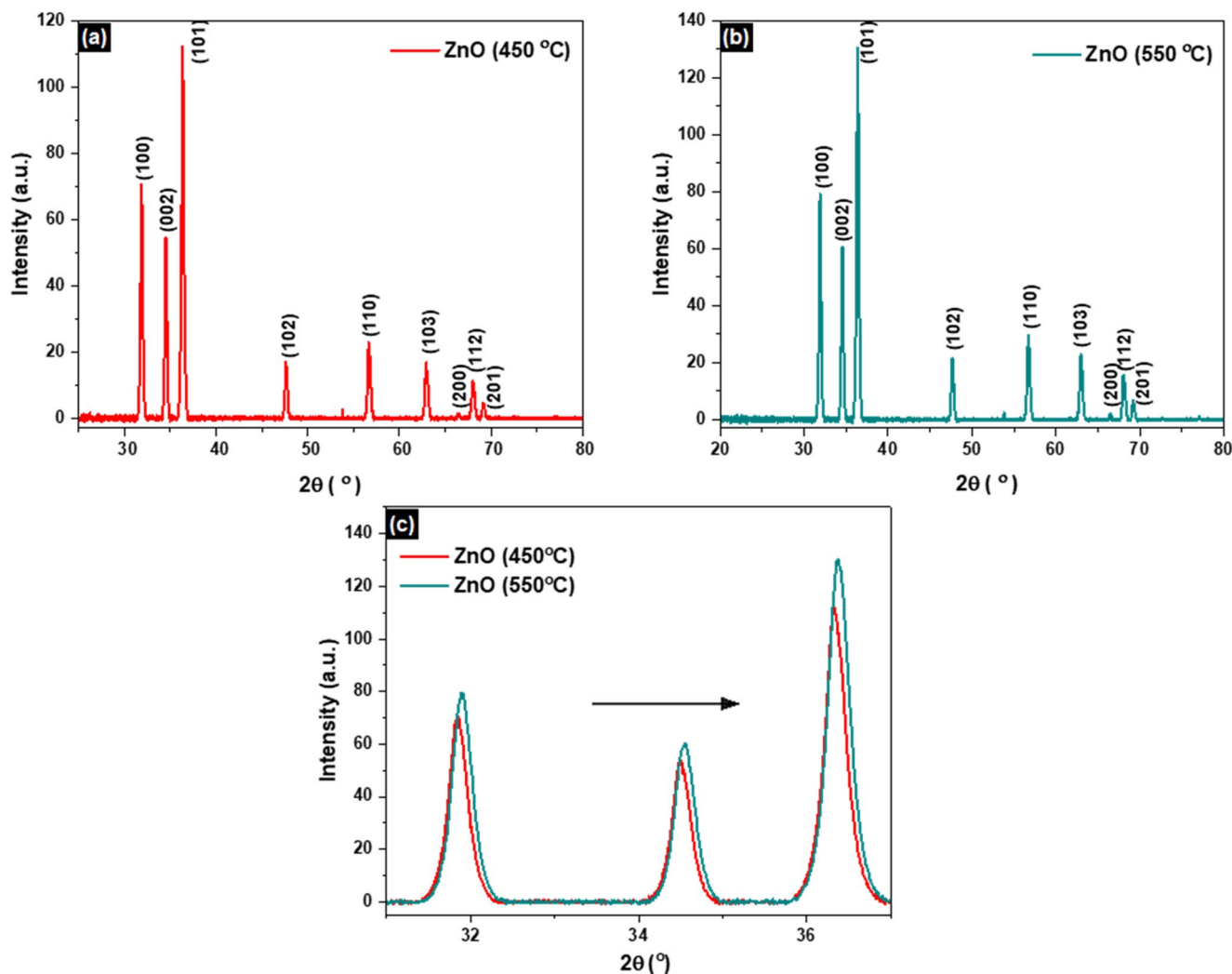


Fig. 1 XRD pattern of the green synthesized ZnO nanoparticles prepared using leaf extract of *Seasea lancea* at different annealing temperatures of (a) 450 °C, and (b) 550 °C. (c) An overlaid lower range diffraction pattern of ZnO NPs prepared at 450 and 550 °C

34.52°, 36.30°, 47.74°, 56.67°, 62.90°, 66.63°, 68.04°, 69.19° and they correspond to the crystal planes of (100), (002), (101), (102), (110), (103), (200), (112), and (201) respectively. The absence of any additional peaks confirmed the high phase purity of the synthesized samples. A noticeable increase in peak intensity with increasing calcination temperature from 450 to 550 °C, in addition to the reduced Full Width at Half Maximum (FWHM) values, indicates enhanced crystallinity and stabilization of the hexagonal ZnO phase [40]. The enhanced crystallinity at higher temperature is attributed to the high thermal energy during the synthesis process, which promotes atom migration to stable lattice sites, reduces structural defects, and facilitates lattice reorganization [41]. The selected peaks that correspond to the (100), (002), and (101) planes in the 2 θ range of 31–38° (Fig. 1c) also display a slight shift towards lower diffraction angles, which is associated with increased lattice strain at higher calcination temperatures [42]. These results indicate that both the temperature exerts a significant influence on the crystalline structure of the nanoparticles [43]. Previous studies have shown that green synthesis provides a simple, effective and eco-friendly route for generating ZnO nanoparticles [43–45], consistently yielding the hexagonal phase due to its superior stability under ambient conditions.

Table 1 presents the structural properties of ZnO NPs calcined at 450 and 550 °C deduced from the XRD patterns. The crystallite size (D) was estimated from Debye-Scherrer's equation (Eq. 3), while the microstrain (ϵ), and dislocation density (ρ_d) were obtained using Eqs. 4 and 5, respectively. As the temperature increases, crystallite size increases from 26.95 nm to 27.36 nm. The lattice parameters ($a=b$ and c) remain nearly unchanged between the two temperatures, suggesting that the wurtzite hexagonal structure is maintained. The minor variations observed may be associated with stress arising from grain growth and native imperfections such as oxygen vacancies, point defects, or dislocations [46, 47]. Additionally, surface effects such as lattice relaxation caused by dangling bonds could contribute to slight lattice distortions, as these bonds interact electrostatically with atmospheric oxygen ions, resulting in partial contraction of the lattice [48]. It is also evident that the crystallinity of the ZnO nanoparticles increases from 92.1 to 94.6% at 450 °C and 550 °C, respectively. These results confirm that higher calcination temperatures favour

enhanced crystallinity and grain coalescence, which reduce structural defects and promote lattice reorganisation.

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (3)$$

$$\epsilon = \frac{\beta}{4\tan\theta} \quad (4)$$

$$\rho_d = \frac{1}{D^2} \quad (5)$$

Where λ is the wavelength of X-ray, k is a constant (0.94), θ is the diffraction angle and, β represent the full width at FWHM of the peak.

Figure 2 illustrates the XRD patterns of the green-synthesised Ag-doped ZnO nanoparticles at different doping concentrations. The diffraction peaks observed at 2 θ values corresponding to the (100), (002), (101), (102), (110), (103), (112), and (201) planes are characteristic of the hexagonal wurtzite structure of ZnO, in agreement with the standard JCPDS card no. 36–1451 [49, 50]. Additional peaks observed at 2 θ values of 40.67°, 44.37°, 66.19°, 72.44°, and 76.98° are assigned to the (111), (200), (220), and (311) planes of face-centred cubic (fcc) silver, as referenced by JCPDS no. 87–0717 [51]. These peaks confirm the presence of metallic Ag as a secondary phase within the ZnO matrix [52]. An increase in the Ag doping ratio leads to an improvement in the intensity of the Ag-related peaks, while the peak shifts slightly to the lower 2 θ values across all Ag/ZnO samples. The extent of Ag incorporation at different concentrations and their influence on the ZnO lattice were compared with the pristine ZnO by plotting the lower angle patterns of the (111), (200), (220), and (311) peaks of Ag/ZnO nanocomposites (Fig. 2b). An increase in Ag doping level caused a noticeable shift of the XRD peaks toward higher 2 θ values, suggesting a correlation between peak position and the degree of Ag incorporation into the ZnO crystal plane [52]. This shift can be ascribed to internal lattice stress arising from the mismatch in ionic radii between Zn²⁺ and the substituting Ag ions [53]. Moreover, the incorporation of Ag into ZnO nanoparticles resulted in a slight decrease in the overall diffraction intensity of Ag/ZnO, reflecting a minor reduction in crystallinity. This effect is attributed to

Table 1 Structural parameters of the green-synthesised ZnO NPs at 450 °C and 550 °C

Sample	Crystallite size (D) (nm)	Interplanar spacing (d) Å	Lattice parameters (Å) a , b , c	Volume ($V/10^6$ pm ³)	Dislocation Density (δ) (nm ⁻²)	Microstrain (ϵ)	Crystallinity (%)
ZnO (450)	26.95	1.95150	$a=b=3.2504$ $c=5.2060$	47.63	0.001401	0.003245	92.1
ZnO (550)	27.36	1.95586	$a=b=3.2523$ $c=5.2072$	47.70	0.001453	0.003177	94.6

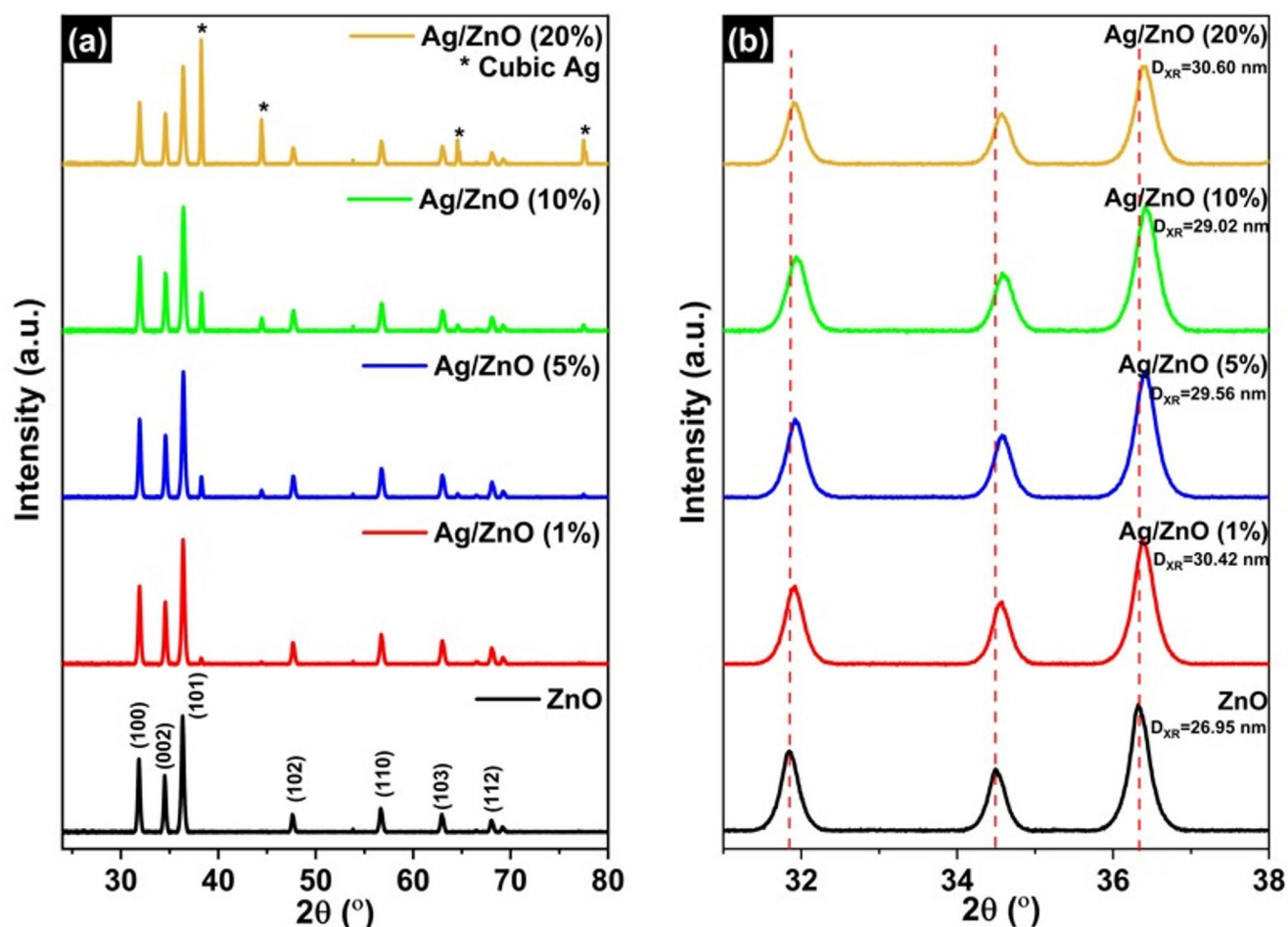


Fig. 2 Overlaid XRD patterns of pristine ZnO and the Ag-doped ZnO at different dopant concentrations in the 2θ range of (a) $25\text{--}80^\circ$, and (b) lower range of $31\text{--}38^\circ$

structural defects and lattice distortions introduced by Ag ions during doping [50, 54]. Table 2 presents the variation of microstructural properties with changes in the amount of the dopant. The crystallite size of the Ag/ZnO nanoparticles was observed to be larger than that of pristine ZnO, indicating that Ag incorporation influences the growth kinetics of ZnO nanoparticles. Similar results have been reported in other studies [55, 56]. The dislocation density and lattice strain increased progressively with Ag doping up to 10%, confirming that Ag incorporation induces lattice distortions

and defect formation within the ZnO crystal structure [57, 58]. However, at 20% doping, both parameters decreased, suggesting a partial relaxation of the lattice.

3.2 Optical properties

The optical and electronic properties of green-synthesized ZnO nanoparticles calcined at 450 and 550 °C, as well as Ag-doped ZnO nanoparticles with 1, 5, 10, and 20% Ag doping, were studied by UV–vis spectroscopy. As seen in Figs. 3a and b, the ZnO nanoparticles prepared at different temperatures displayed distinct absorption edges around 380 nm, which align with the characteristic absorption band of ZnO associated with the wurtzite crystal structure, typically reported in the 350–380 nm region. A slight redshift in the absorption maximum was observed with increasing temperature [59], which can be attributed to the partial growth of crystallite size.

The Ag-doped ZnO samples exhibited a strong and sharper absorption edge (350–420 nm), confirming the enhanced photon absorption of ZnO upon Ag incorporation

Table 2 Structural parameters of Ag-doped ZnO with different dopant %

Sample	Crystallite size (D) (nm)	Interplanar spacing (d) (Å)	Dislocation Density (ρ_d) (nm^{-2})	Microstrain (ϵ)
Ag/ZnO (1%)	30.42	1.9443	1.297×10^{-3}	3.030×10^{-3}
Ag/ZnO (5%)	29.56	1.9469	1.310×10^{-3}	3.086×10^{-3}
Ag/ZnO (10%)	29.02	1.8868	1.316×10^{-3}	3.048×10^{-3}
Ag/ZnO (20%)	30.60	1.8874	1.167×10^{-3}	2.848×10^{-3}

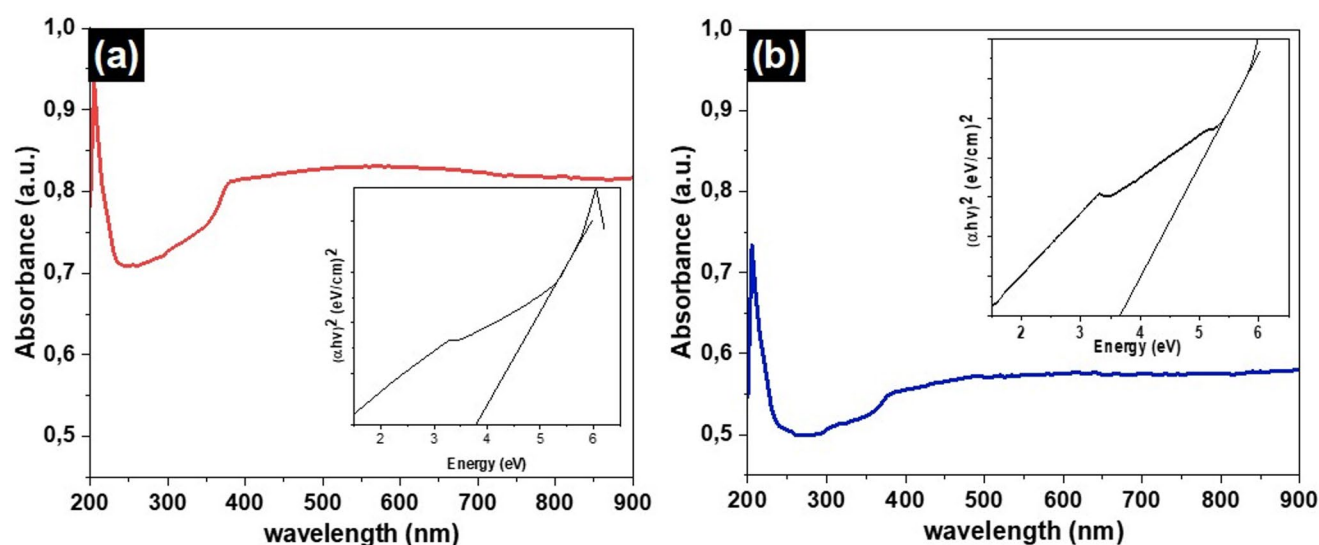


Fig. 3 (a) Absorption spectra of the green synthesised ZnO nanoparticles prepared from the extract of *Seasea lancea* at (a) 450 °C and (b) 550 °C, with their respective Tauc plot in the inset

[57]. A redshift of the absorption edge towards longer wavelengths was observed with increasing Ag content, demonstrating improved visible-light responsiveness of the samples. In addition, the absorption intensity in the visible region (400–800 nm) increased significantly due to the localized surface plasmon resonance (LSPR) effect of Ag, which is expected to enhance the photocatalytic performance of the nanomaterials [60]. The observed shift in the absorption peak indicates a modification of the optical band gap (E_g) resulting from Ag doping.

The optical band gaps (E_g) of the nanomaterials were estimated using the Tauc relation presented in Eq. 6 [61], whereby $(\alpha h\nu)^2$ was plotted against the photon energy ($h\nu$). The band gap energy of the pristine ZnO prepared at 450 and 550 °C was found to be 3.86 and 3.65 eV, respectively. With Ag doping, the band gap energies decreased progressively to 3.56, 3.50, 3.32, and 3.24 eV for 1, 5, 10, and 20% doping levels, respectively, as shown in Figs. 4(a–c). This could be attributed to the creation of oxygen vacancies, which promote electronic transitions from the valence band (VB) to the conduction band (CB) [62]. In addition, Ag acts as an acceptor in the ZnO lattice, introducing intermediate states in the band gap that narrow the band gap energy. This is due to the emergence of the p-type conductivity in the Ag-doped ZnO, where the acceptor states facilitate the formation of an impurity band [63, 64]. In contrast to a similar study, a slight increase in the band gap energy has been reported at high doping concentration. This was ascribed to grain coalescence and reduced defects, which suppressed the intermediate states responsible for band gap narrowing [65, 66].

$$(\alpha h\nu)^n = A(h\nu - E_g) \quad (6)$$

where α represents the absorption coefficient, ν is the light frequency, A is a constant, h is Planck's constant, E_g is the optical band gap, and $n=2$ for direct transitions and $n=1/2$ for indirect transitions.

3.3 Functional groups analysis

The functional groups present in the pristine and doped ZnO were studied using FTIR spectroscopy, and Fig. 5 presents the results of the spectral analysis. The FTIR spectra exhibit characteristic absorption features confirming the formation of the oxide framework and the influence of silver incorporation. In the spectrum of pristine ZnO, an absorption band below 600 cm^{-1} was observed, corresponding to the Zn–O stretching vibration, which is the fingerprint of the wurtzite ZnO lattice. The Ag-doped ZnO samples retained this Zn–O band, but with slight shifts in band position and variations in intensity, suggesting subtle modifications of the local bonding environment due to Ag incorporation into the ZnO lattice. Weak bands around 2966 and 2897 cm^{-1} can be ascribed to –CH bonds [67]. An intense peak at 2356 cm^{-1} can be attributed to the $\text{C}\equiv\text{C}$ and C–N functional groups or to atmospheric CO_2 absorbed during the analysis [68]. In addition, other peaks at 1447, 889, and 537 cm^{-1} may be assigned to C–H bending vibration, =C–H bending vibration, and the stretching vibration mode of Zn–O bond, respectively [69, 70]. The absence of distinct new bands attributable to Ag–O vibrations suggests that silver ions are either substituted into the Zn^{2+} lattice sites or dispersed as highly reduced metallic Ag without forming separate Ag oxide phases. Overall, the comparative FTIR analysis confirms that Ag doping preserves the structural

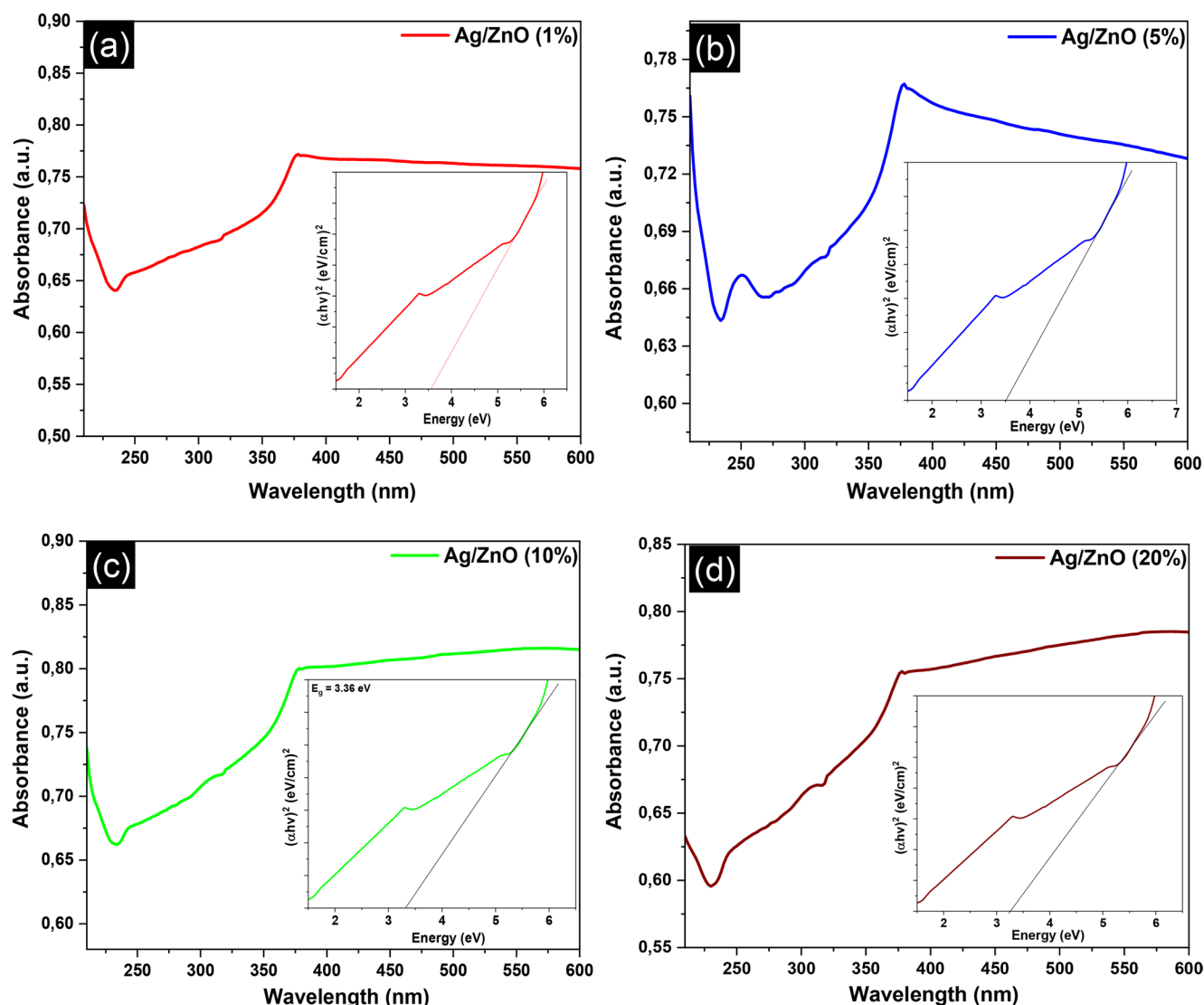


Fig. 4 UV-vis spectra of Ag/ZnO at (a) 1, (b) 5, (c) 10, and (d) 20% Ag doping concentration, and their respective Tauc plots as insets

integrity of ZnO while inducing local modifications in the vibrational environment, which are consistent with the successful incorporation of Ag species. These observations are comparable to previous studies [71, 72].

3.4 Morphological analysis

Figure 6 presents the SEM images of the ZnO NPs prepared at 450 and 550 °C, which explore their surface morphology. The figure shows that the sample prepared at 450 °C displayed clear spherical grains with well-defined boundaries and minor agglomerations, indicating a relatively uniform morphology. As the calcination temperature increased to 500 °C, the nanoparticles displayed a more pronounced agglomeration (Fig. 6b), which resulted in an increase in grain size. This observation was further supported by the

grain size distribution histogram (Fig. 6c) derived from the SEM images. The results showed that the ZnO nanoparticles calcined at 450 °C had an average grain size of approximately 2.32 μm , while those prepared at 550 °C exhibited a larger average size of about 2.56 μm . The increase in grain size with calcination temperature could be attributed to enhanced atomic diffusion and particle agglomeration at higher energy, which promotes grain growth [73].

SEM micrographs of the Ag-doped ZnO samples (Fig. 7a-d) revealed predominantly interconnected spherical particles that formed a compact structure. Noticeable changes were observed as silver doping increased. Progressive agglomeration was observed from 10% to 20% doping, with the 20% sample exhibiting distinct hollow regions, indicating that higher levels of doping promote particle clustering. A similar trend with increasing dopant concentration

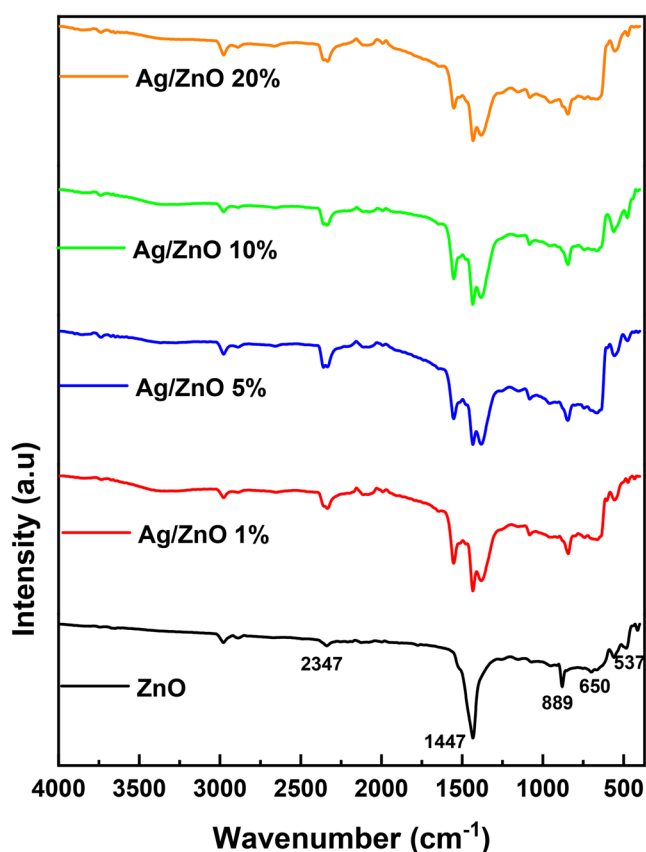


Fig. 5 Overlaid FTIR spectra of the pristine ZnO and Ag-doped ZnO nanoparticles at different percentage dopants

was reported in studies by Sah et al. [74]. The observation was further supported by the grain distribution histogram (Fig. 7(e–h)), which displays the average particle size of the Ag-doped ZnO as 2.01, 2.15, 1.88, and 2.40 μm for 1, 5, 10, and 20% doping, respectively.

3.5 Photocatalytic degradation of methyl orange

The green-synthesized ZnO and Ag-doped ZnO were investigated as catalysts for the degradation of methyl orange (MO) under the irradiation of visible light. The photocatalytic activity was evaluated for 120 min by recording the absorbance at the characteristic maximum wavelength of methyl orange (464 nm). As shown in Fig. 8, the degradation of MO under visible light irradiation alone achieved 43.67%, confirming the limited efficiency of direct photolysis. In the presence of a pristine ZnO catalyst, degradation efficiency increased substantially to 61.35% (Fig. 8a). Upon the utilization of Ag-doped ZnO photocatalysts, a marked enhancement in degradation efficiency was achieved. After 120 min of visible light irradiation Ag/ZnO (1%), Ag/ZnO (5%), Ag/ZnO (10%), and Ag/ZnO (20%) exhibited degradation efficiencies of 93.20%, 98.06%, 99.35%, and 95.85.42% as presented in Fig. 8b, c and d, and 8e, respectively. The outstanding activity of Ag-doped

ZnO can be ascribed to its reduced band gap energy, which facilitates more effective charge separation while suppressing electron–hole recombination. In addition, the enhanced optical absorption of Ag/ZnO in the UV–vis region, as verified by spectroscopic analyses, further corroborates its superior photocatalytic performance compared to pristine ZnO and other doping concentrations [75].

The kinetics of MO degradation were evaluated using the 1st-order model (Fig. 9a), and the oxidation rate constants (K) were obtained from the linear fits of $-\ln(C/C_0)$ versus time. The correlation coefficients (R^2) ranged from 0.939 to 0.996, confirming that the advanced oxidation of MO under the study conditions follows pseudo-1st-order kinetics. The calculated K values were 0.00235, 0.00773, 0.01247, 0.01773, 0.03336 and 0.00907 min^{-1} for photolysis, ZnO, Ag/ZnO (1%), Ag/ZnO(5%), Ag/ZnO(10%), and Ag/ZnO(20%), respectively. These results highlight the negligible self-oxidation under visible light irradiation alone (photolysis). Ag/ZnO (10%) exhibited the highest rate constant, nearly 4.3 times higher than pristine ZnO. The enhanced photocatalytic degradation can be attributed to the synergistic effect of Ag nanoparticles, which promote charge separation, extend the absorption range, and suppress electron-hole recombination. Further increase in Ag content to 20% resulted in a slight decrease in the rate constant, which could be attributed to excessive Ag nanoparticles acting as recombination centres as well as blocking surface active sites. The results obtained in this study were compared with previously reported studies on the photodegradation of MO using Ag–ZnO NPs. To further evaluate the broad applicability of the optimal 10% Ag/ZnO catalyst, its performance was investigated for the degradation of crystal violet dye and ibuprofen. As illustrated in Fig. 9b, the catalyst exhibited significant activity toward both contaminants, achieving 84.99% and 83.34% degradation efficiencies for crystal violet dye and ibuprofen, respectively.

The degradation efficiency achieved by the green-synthesized Ag–ZnO NPs in the current study is comparable to, and in some cases exceeded, the results obtained from using chemically synthesised Ag-doped ZnO nanoparticles (Table 3), demonstrating the effectiveness of the synthesis approach. The bioactive compounds from *Searsia lancea* leaf extract may have contributed to more uniform nanoparticle formation with increased surface area, providing more sites for photocatalytic degradation [84].

4 Conclusion

In this study, Ag-doped ZnO photocatalysts were successfully synthesized via a simple green fabrication route using *Searsia lancea* leaf extract, which acted as both a reducing and

Fig. 6 SEM micrographs of ZnO nanoparticles calcined at (a) 450 °C and 500 °C and grain size distribution histograms derived from the SEM images for (c) ZnO calcined at 450 °C and (d) ZnO calcined at 500 °C

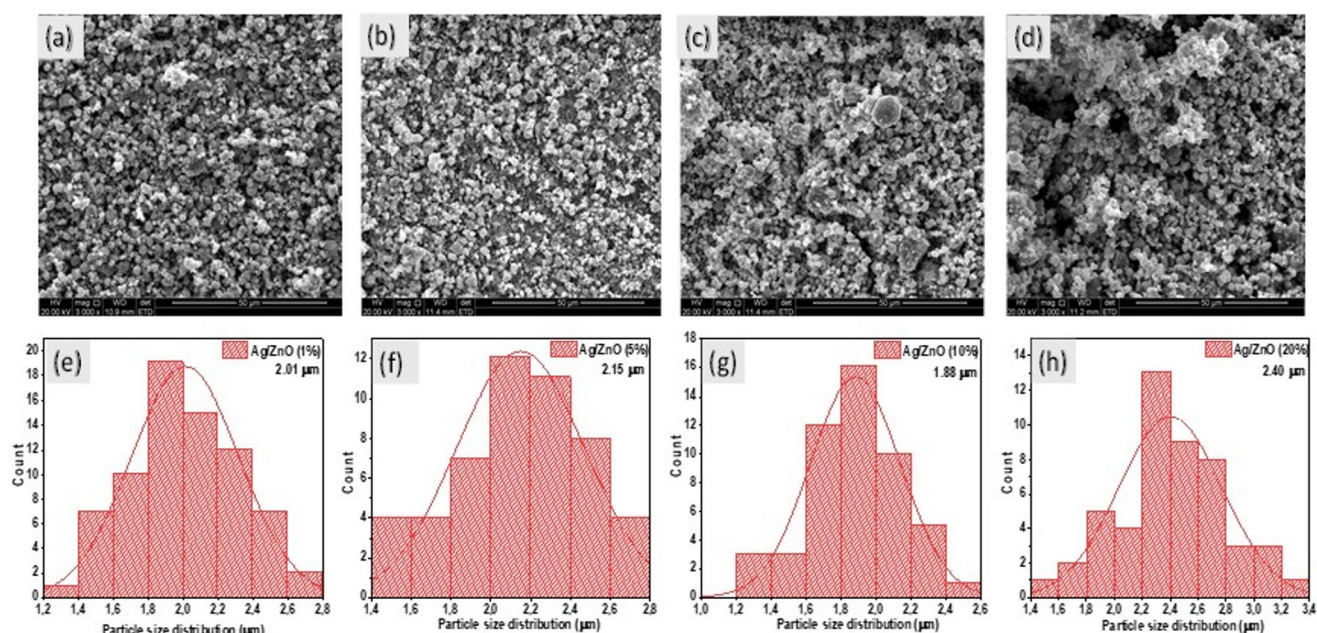
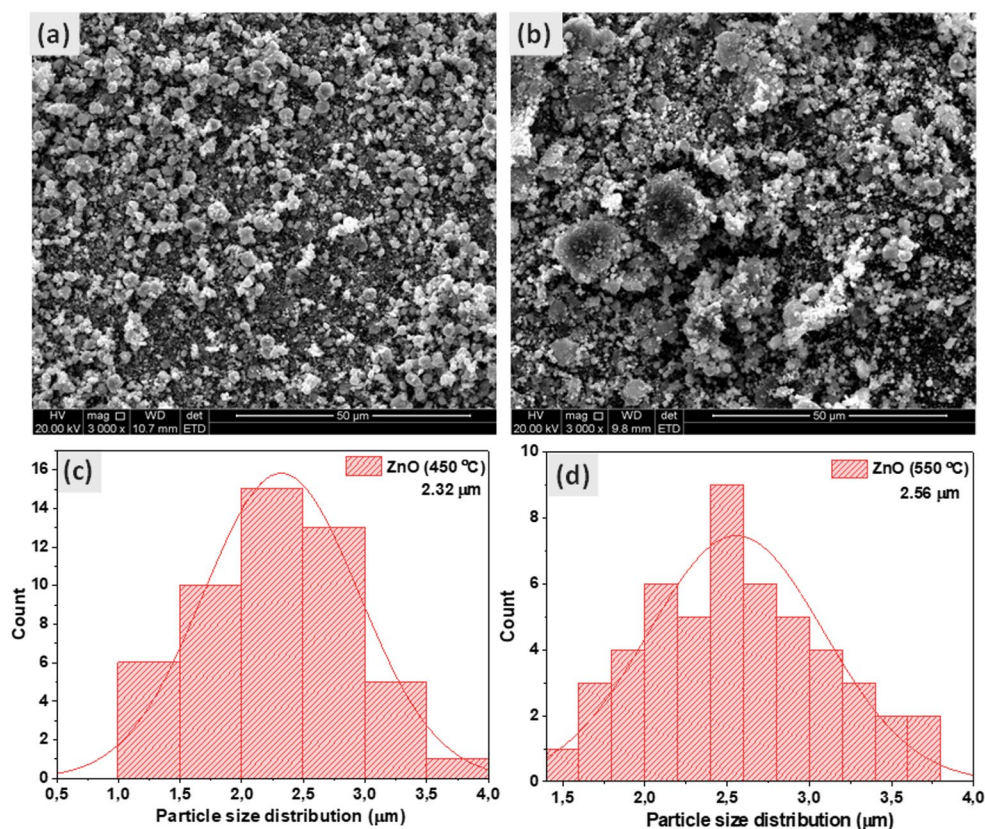


Fig. 7 SEM micrographs of Ag-doped ZnO nanoparticles at different doping levels (a) 1%, (b) 5%, (c) 10% and (d) 20%; corresponding grain size distribution histograms derived from the SEM images for (e) 1%, (f) 5%, (g) 10% and (h) 20%

stabilising agent. The introduction of Ag into the ZnO lattice progressively reduced the band gap, decreasing from 3.86 eV for pristine ZnO to 3.56, 3.50, 3.32, and 3.24 eV at doping levels of 1, 5, 10, and 20%, respectively, thereby highlighting a

substantial alteration in the electronic structure of the material. All the Ag-doped ZnO samples exhibited good MO removal efficiency, with Ag/ZnO (10%) composite showing the highest MO degradation efficiency of 99.3%. The improved

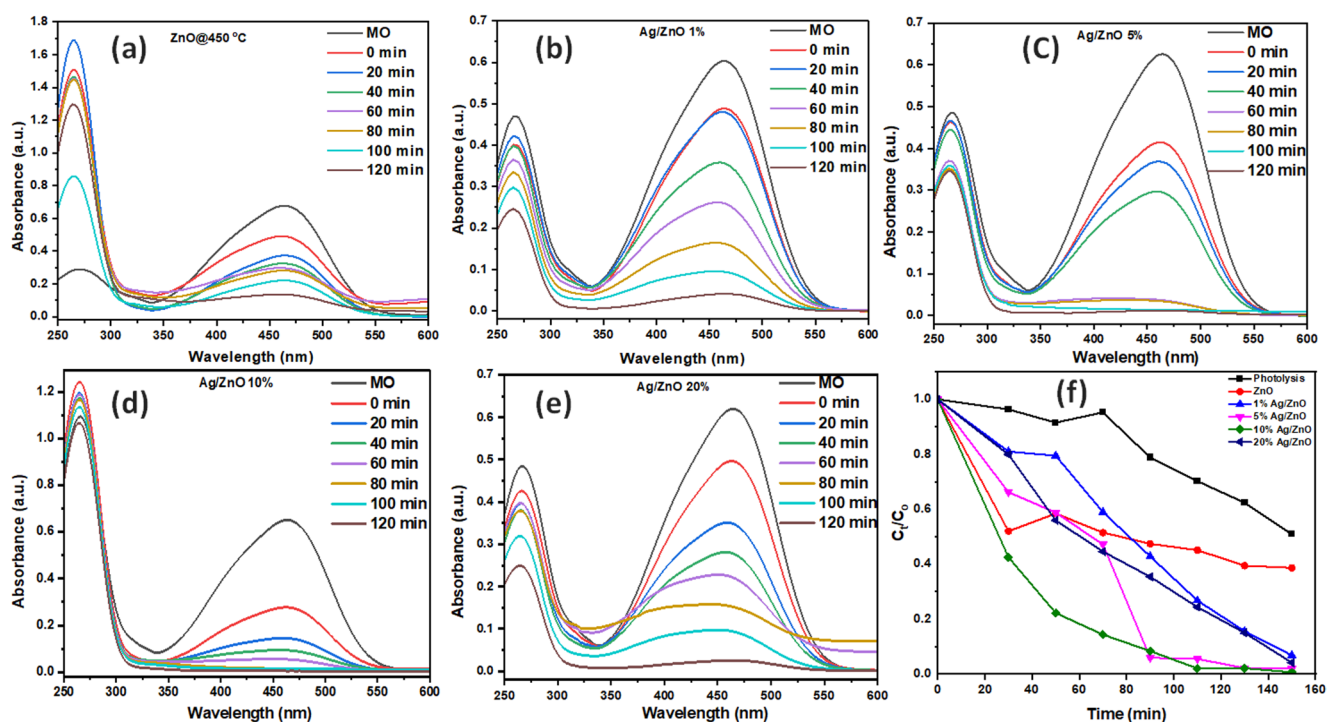


Fig. 8 UV-vis absorption profiles of methyl orange (MO) during photocatalytic degradation under (a) photolysis, (b) ZnO, and (c–f) Ag-doped ZnO at 1%, 5%, 10% and 20% doping levels, (g) Overall degradation profiles of MO comparing photolysis, pure ZnO, and Ag-doped ZnO catalysts

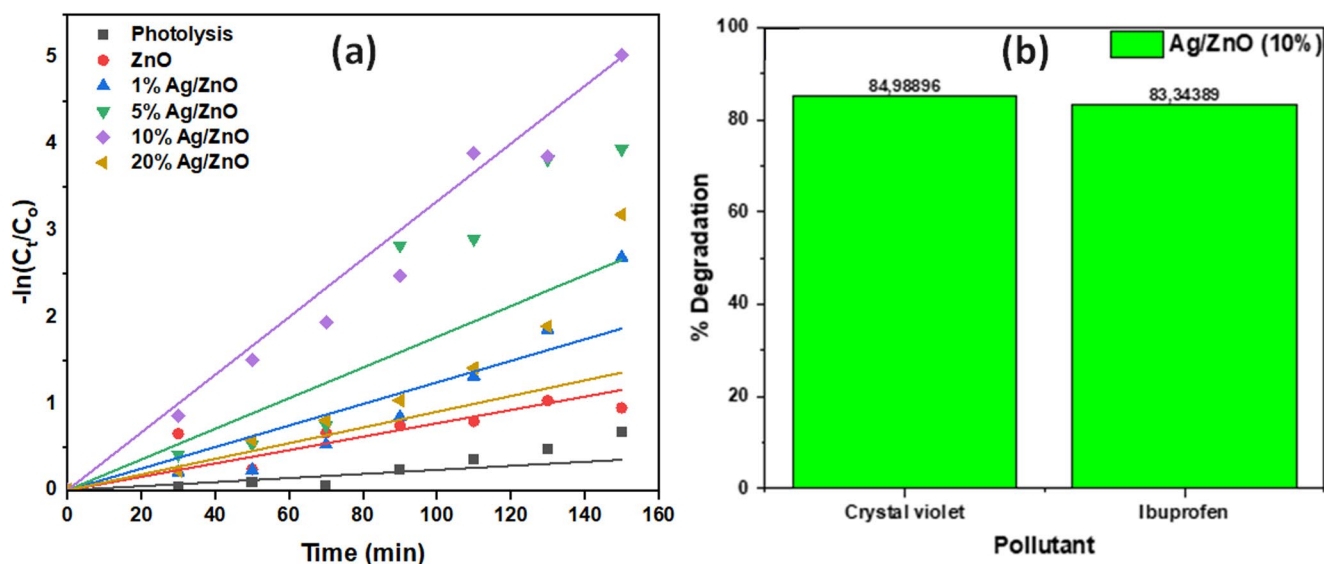


Fig. 9 (a) Pseudo first order kinetics plots for the photodegradation of methyl orange under photolysis, pristine ZnO, and Ag-doped ZnO (1%, 5%, 10% and 20%), (b) Evaluation of the broad applicability of

the optimal 10% Ag/ZnO catalyst for the photodegradation of crystal violet dye and ibuprofen

photocatalytic degradation results from the synergistic effect of Ag nanoparticles, which enhance charge separation, broaden the absorption spectrum, and inhibit electron-hole recombination. The bioactive compounds from the leaf extract also aid in the uniform formation of nanoparticles with a larger surface area, thereby increasing the availability of active sites for photocatalytic degradation. The performance of the optimal Ag/

ZnO (10%) catalyst was also evaluated for the degradation of other pollutants, including crystal violet dye and ibuprofen, achieving efficiencies of 84.9% and 83.3%. Overall, this study presents an eco-friendly approach that allows the continued use of dyes while significantly mitigating adverse effects, protects water resources, and contributes to the United Nations Sustainable Development Goals.

Table 3 A comparison of MO degradation by the prepared Ag-doped ZnO was reported in this study with other similar studies in the literature

Nanomaterials	Synthesis method	Ag loading	Catalyst dosage/MO concentration/ Time/pH	Degradation Efficiency (%)	Rate Constant (min ⁻¹)	Ref
Ag–ZnO NPs	Co-precipitation (Zn(NO ₃) ₂ + KOH, SDS as capping agent)	1.0 mol%	0.5 g/L catalyst; 10 mg/L MO; 120 min; pH ~ 6.5	96.74	0.031 (pseudo-first-order)	[76]
Ag–ZnO NPs	Co-precipitation	4 wt%	0.15 g/L catalyst; 10 mg/L MO; solution; 120 min; pH (neutral range)	86.5	0.0147 (pseudo-first-order)	[77]
Ppy/Ag–ZnO nanocomposite	Green mechanochemical (ball milling + AgNO ₃ + Ppy)	4.5 mol%	2 mg catalyst; 5 ppm MO; 90 min	91.11	0.028 (pseudo-first-order)	[78]
Ag/ZnO/Graphene nanocomposite	One-step microwave-assisted solution synthesis	1 wt%	50 mg catalyst; 20 ppm MO; 80 min	99.6	0.05182 (pseudo-first-order)	[79]
GO-loaded 1 mol% Ag/ZnO	Green precipitation using medicinal plant extract	1 mol%	100 mg catalyst; 20 ppm; 210 min	27.46	-	[80]
Ag-ZnO (AZ-1)	Co-precipitation	1% Ag	100 mg catalyst; 10 ppm MO; 30–120 min	92	-	[81]
Ag-ZnO nano-spindles (AZ4)	Wet chemical method	-	5 mg catalyst; 10 µM MO; 20–40 min	88	-	[82]
Ag-ZnO nanoparticles	Green route via <i>Withania somnifera</i> root extract	-	5 mg catalyst; 3 mL 10 ⁻⁵ M MO; 11 min	95	Pseudo-first-order	[83]
Ag-ZnO nanoparticles	Green synthesis using <i>Searsia lancea</i> leaf extract	10% wt%	20 mg catalyst; 10 mg/L MO; 150 min.	99.3	0.03336 (pseudo-first-order)	Current study

Author contribution KAM: Writing - Original draft preparation, Formal analysis, Methodology. LS: Investigation, Writing - Review & Editing. NHR: Supervision, Writing - Review & Editing. CA: Resources, Writing - Review & Editing. DCO: Conceptualisation, Supervision, Resources, Writing - Review & Editing.

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Data availability Data is provided within the manuscript.

Declarations

Conflict of interest The authors declare no conflict of interest.

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