

Sustainable Green Synthesis of ZnO Photocatalysts Using Shallot Peel Extract as Capping Agent for Efficient Photodegradation of Rhodamine B

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Abstract

Green synthesis strategies offer a sustainable approach for preparing semiconductor photocatalysts with tunable properties. In this study, ZnO particles were synthesized using shallot peel extract, and their structural, morphological, optical, and photocatalytic properties were systematically investigated. Structural characterization by Raman spectroscopy and X-ray diffraction confirmed the formation of phase-pure wurtzite ZnO, with crystallite sizes in the nanometer range. Scanning electron microscopy revealed hexagonal rod-like morphologies with dimensions of 1–2 μm , which are interpreted as polycrystalline assemblies of aggregated nanocrystallites. Optical analysis using UV–Vis diffuse reflectance spectroscopy showed a direct bandgap of 3.02 eV and additional visible-light absorption, which may be associated with defect states and possible extrinsic contributions. Photoluminescence spectroscopy exhibited green emission attributed to intrinsic defects. The photocatalytic performance of the synthesized ZnO was evaluated through the removal of Rhodamine B under irradiation, achieving a maximum removal efficiency of 97.68% within 150 min. Kinetic analysis indicated that the process follows a pseudo-first-order model with an apparent rate constant of 0.0290 min^{-1} . The observed photocatalytic performance may be associated with the structural and optical characteristics of the synthesized ZnO. However, the specific roles of these factors, as well as the contribution of adsorption, cannot be conclusively distinguished in the absence of a control sample. Overall, this study provides insight into the relationship between synthesis conditions and the resulting properties of green-synthesized ZnO, while highlighting the need for further investigation to clarify the underlying mechanisms. This work demonstrates that shallot peel extract is an effective and environmentally benign capping agent for engineering ZnO photocatalysts with high efficiency for wastewater treatment applications.

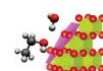
Keywords: Green synthesis; Shallot peel extract; Photocatalysis; Rhodamine B; Zinc oxide.

1 Introduction

The rapid expansion of industrial activities has led to the widespread release of organic dye pollutants into aquatic environments, posing serious threats to ecosystems and human health [1]. Synthetic dyes such as Rhodamine B are widely used in textile, printing, and pharmaceutical industries and are known for their high stability, toxicity, and resistance to conventional wastewater treatment processes [2]. Among advanced treatment technologies, semiconductor-based photocatalysis has attracted

significant attention due to its ability to mineralize organic pollutants into harmless end products using light as a clean energy source [3,4]. However, the practical application of photocatalysis is still limited by the availability of efficient, stable, and environmentally benign photocatalytic materials.

Zinc oxide (ZnO) is a widely studied semiconductor photocatalyst owing to its suitable bandgap, strong oxidation ability, low cost, and chemical stability [5,6]. With a direct bandgap of approximately 3.2 eV, ZnO exhibits excellent



photoactivity under ultraviolet irradiation and has been extensively investigated for dye degradation and environmental remediation [3,6,7]. Nevertheless, the photocatalytic efficiency of ZnO is often hindered by rapid electron-hole recombination and limited visible-light absorption [8]. Various strategies, including defect engineering, morphology control, and surface modification, have therefore been explored to enhance its photocatalytic performance [8,9].

In recent years, green synthesis approaches have emerged as sustainable alternatives to conventional chemical routes for synthesis of material [10,11]. Plant extracts are particularly attractive as they contain natural bioactive compounds that can act as reducing, stabilizing, and capping agents, thereby enabling controlled crystal growth while minimizing environmental impact. Shallot and red onion peel extract, an abundant agricultural waste, is rich in polyphenols and anthocyanins that can interact with metal oxide surfaces and influence nucleation and growth processes [12–14]. Despite its potential, the use of shallot peel extract for tailoring the structural and defect properties of ZnO photocatalysts remains relatively unexplored.

In this work, ZnO photocatalysts were synthesized via a sustainable green synthesis route using shallot peel extract as a bio-derived agent. Rather than focusing solely on the use of plant extract, this study emphasizes the relationship between synthesis conditions and the resulting material properties. The structural, morphological, and optical characteristics of ZnO—including crystallite features, polycrystalline hexagonal rod-like morphology, and defect-related optical behavior—were systematically analyzed using Raman spectroscopy, X-ray diffraction, electron microscopy, UV-Vis diffuse reflectance spectroscopy, and photoluminescence analysis. The photocatalytic performance was evaluated through the degradation of rhodamine B, with kinetic behavior described using a pseudo-first-order model. By correlating these structural and optical characteristics with photocatalytic activity, this work provides insight into the structure-property-performance relationship in green-synthesized ZnO systems, particularly in relation to defect-associated visible-light response.

2 Method

2.1 Material

Shallot peel was collected from Kefamenanu traditional market. Zinc acetate dihydrate

($\text{Zn}(\text{CH}_3\text{COO})_2$) and sodium hydroxide (NaOH) was purchased from Merck.

2.2 Preparation of Shallot Peel Extract

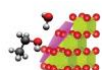
The Shallot peels were first ground using a blender and subsequently sieved to separate coarse and fine powders. The extraction process was carried out using a maceration method. Briefly, 100 g of the fine shallot peel powder was mixed with 1 L of distilled water and allowed to soak at room temperature for 24 h. The resulting mixture was then filtered to remove solid residues, yielding a concentrated extract. This concentrated shallot peel extract was subsequently used as a capping agent in the synthesis of ZnO particles.

2.3 Synthesis of ZnO

A total of 2.64 g of zinc acetate dihydrate was dissolved in 150 mL of distilled water. Subsequently, 2.5 mL of shallot peel extract was added to the solution. The resulting mixture was stirred using a magnetic stirrer for 30 minutes at room temperature. Then, 30 mL of 0.5 M NaOH solution was slowly added dropwise into the mixture under continuous magnetic stirring. The mixture was tightly sealed and subjected to a reaction at 100 °C for 2 hours using an oil bath. After completion of the reaction, the reaction vessel was allowed to cool to room temperature. The resulting precipitate was separated by centrifugation and washed several times with distilled water and ethanol. The obtained solid was then dried in an oven at 100 °C for 2 hours, followed by calcination at 400 °C for 4 hours.

2.4 Characterization

The structural properties of the synthesized particles were examined by Raman spectroscopy and powder X-ray diffraction (XRD). Raman measurements were performed using a Bruker Senterra Raman spectrometer, while XRD patterns were recorded on a Rigaku MiniFlex diffractometer employing Cu K α radiation. The crystallite sizes of the samples were estimated using the Scherrer equation. Particle morphology was investigated by scanning electron microscopy (SEM) using a Hitachi SU3500 instrument operated at an accelerating voltage of 10 kV. Nitrogen physisorption measurements were carried out using a Quantachrome analyzer to evaluate the textural properties of the synthesized particles. The specific surface area and pore size distribution were calculated using the Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) methods, respectively. Optical



properties were analyzed by UV–Vis diffuse reflectance spectroscopy (DRS) using an Evolution 220 spectrometer. The optical bandgap energies were determined from Tauc plots derived from the Kubelka–Munk function, assuming a direct bandgap transition. Photoluminescence (PL) spectroscopy was employed to investigate defect-related emission behavior and luminescence characteristics of the ZnO samples. PL measurements were conducted at room temperature using UV LED excitation with a peak wavelength of 371 nm.

2.5 Photocatalytic Activity Evaluation

The photocatalytic activity of the synthesized ZnO was evaluated through the photodegradation of Rhodamine B (RhB), which was employed as a model organic dye pollutant. In a typical experiment, 50 mg of ZnO nanoparticles was dispersed in 50 mL of RhB aqueous solution with an initial concentration of 5 ppm. Prior to light irradiation, the suspension was placed in a photocatalytic reactor and magnetically stirred in the dark for 30 min to establish adsorption–desorption equilibrium. After this dark equilibration period, a 5 mL aliquot was withdrawn. Subsequently, UV–Vis light irradiation was initiated while continuous magnetic stirring was maintained. At regular time intervals of 30 min, a 5 mL aliquot was collected. This procedure was repeated for a total irradiation time of 150 min, calculated from the onset of light exposure. The photocatalyst particles were separated from the reaction solution by centrifugation, and the resulting supernatant was

analyzed using UV–Vis spectroscopy to determine the residual RhB concentration.

The photodegradation kinetics were analyzed using a pseudo-first-order kinetic model, expressed in Eq. 1.

$$\ln\left(\frac{C_t}{C_0}\right) = -kt \quad (\text{Equation 1})$$

where C_t is the RhB concentration at reaction time t , C_0 is RhB concentration at $t=0$, t is the irradiation time, and k is the apparent rate constant. The value of k was obtained from the slope of the linear plot of $\ln\left(\frac{C_t}{C_0}\right)$ vs t .

The photocatalytic degradation efficiency (η) was calculated using Eq. 2.

$$\eta(t) = \frac{C_0 - C_t}{C_0} \times 100\% \quad (\text{Equation 2})$$

where $\eta(t)$ represents the degradation efficiency at reaction time t .

3 Result and Discussion

3.1 Structure

The structural properties of the synthesized particles were investigated using Raman spectroscopy and X-ray diffraction (XRD). The results are exhibited in **Fig. 1**. Raman spectroscopy was employed to examine the local structural features of the material. The Raman spectrum of the synthesized sample (**Fig. 1a**) displays characteristic bands that are consistent with those of ZnO. Two prominent peaks located at approximately 100 and 439 cm^{-1} are assigned to the E_{2L} and E_{2H} vibrational modes, respectively, which are characteristic of wurtzite-structured ZnO. In addition, the band observed around 330 cm^{-1} can be attributed to the $E_{2H}-E_{2L}$ multiphonon vibration mode. The E_{2H} mode is associated with

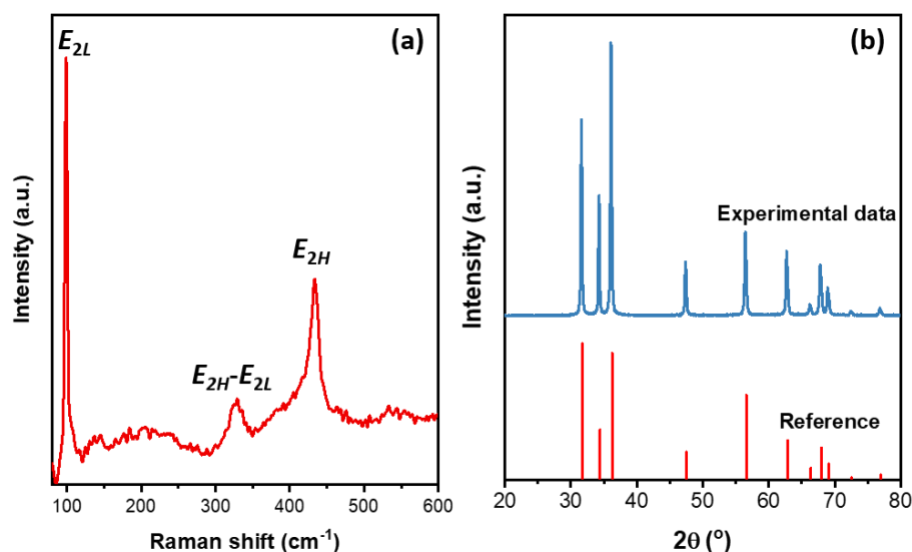
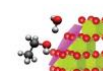


Figure 1. Structural characterization of synthesized sample; (a) Raman spectra; (b) XRD pattern.



vibrations of oxygen atoms, whereas the E_{2L} mode arises from vibrations of zinc atoms within the ZnO crystal lattice [15]. Notably, the broadening of the E_{2H} mode suggests a relatively small crystallite size, indicating that the presence of shallot peel extract effectively suppresses ZnO crystal growth during the synthesis process.

XRD analysis was subsequently carried out to determine the crystallographic structure and phase purity of the synthesized particles. The XRD pattern of the sample, together with the corresponding CIF reference file No. 2300450, is presented in **Fig. 1b**. The diffraction peaks show excellent agreement with the reference data, confirming that the synthesized material is ZnO with a wurtzite crystal structure and a $P6_3mc$ space group (16). This assignment is supported by the presence of three dominant reflections at 2θ values of approximately 31.59° , 34.26° , and 36.08° , which correspond to the (100), (002), and (101) crystallographic planes, respectively [16]. Additional reflections with lower intensities are also observed and closely match those of the reference pattern. The absence of impurity-related peaks indicates that the synthesized ZnO exhibits high phase purity.

Moreover, crystallite size estimation using the Scherrer equation yielded values of 69.51, 70.56, and 65.10 nm for the [100], [002], and [101] planes, respectively. These values are presented as plane-specific estimates without averaging, as they reflect coherent diffraction domain sizes along different crystallographic directions. It should be noted that instrumental broadening was not corrected, and thus the reported values are considered semi-quantitative. Furthermore, the relatively large crystallite sizes do not necessarily contradict the observed broad Raman features, as defect density is governed not only by crystallite

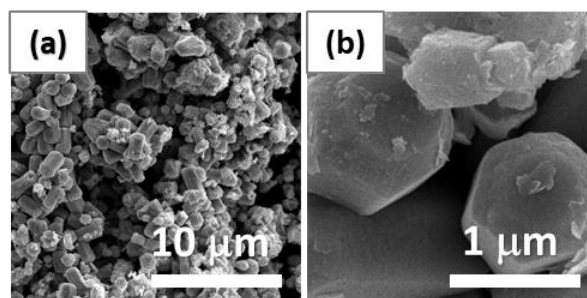


Figure 2. SEM images of the synthesized particle at different magnification; (a) low magnification, (b) high magnification.

size but also by surface states, structural disorder, and synthesis conditions.

3.2 Morphology and Porosity

The morphology of the synthesized ZnO particles was examined using scanning electron microscopy (SEM). **Fig. 2** presents SEM images of the synthesized particles acquired at low and high magnifications. At low magnification (**Fig. 2a**), the particles predominantly exhibit a homogeneous hexagonal rod-like morphology. The high-magnification SEM image (**Fig. 2b**) provides a clearer view of the particle shape and dimensions, revealing well-defined hexagonal structures with diameters in the range of 1–2 μm .

Notably, this particle size is significantly larger than the crystallite size ($\sim 65\text{--}70\text{ nm}$) estimated from XRD analysis, indicating that the microrod structures are not single crystals but are composed of aggregated nanocrystallites. These structures are therefore interpreted as polycrystalline assemblies formed through the aggregation of smaller coherent domains.

In the presence of shallot peel extract, phytochemical components in the extract may interact with the growing ZnO nuclei during synthesis, potentially influencing crystal growth

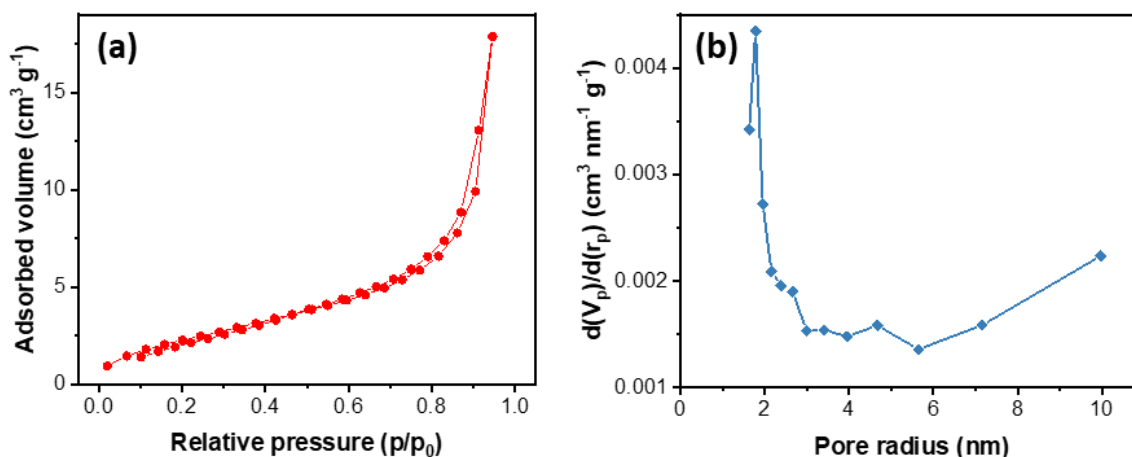
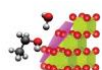


Figure 3. (a) N₂ physisorption isotherm, and (b) BJH pores size distribution of the synthesized particle.



behavior. However, in the absence of a control sample, it is not possible to conclusively determine the specific role of these species or their interaction with particular crystallographic facets (e.g., (0002)) in directing anisotropic growth. Therefore, the observed rod-like morphology is discussed as being associated with the synthesis conditions and is consistent with previous reports, rather than being directly attributed to a facet-specific capping mechanism [17,18]. A comparative study using a control sample would be necessary for a rigorous evaluation of its influence, and this will be considered in future work.

Nitrogen physisorption analysis was carried out to evaluate the specific surface area and pore characteristics of the synthesized particles. The N_2 physisorption isotherm of the sample is shown in **Fig. 3a**. The isotherm can be classified as Type III, indicating weak interactions between N_2 molecules and the ZnO surface. This type of isotherm is typically associated with non-porous or macroporous materials, where adsorption occurs mainly through intermolecular clustering rather than strong adsorbate-adsorbent interactions. The presence of an H3-type hysteresis loop suggests the existence of slit-shaped pores, which are commonly attributed to interparticle voids formed by the aggregation of plate-like or irregular particles, rather than well-defined porous frameworks [19].

Fig. 3b shows the pore size distribution derived using the Barrett–Joyner–Halenda (BJH) method. The distribution indicates the presence of pores in the mesoporous range; however, these pores are more likely associated with interparticle voids rather than intrinsic porosity. It should be noted that the BJH method is not reliable for accurately determining micropore size distribution, and

therefore no conclusions regarding microporosity are drawn in this study. Based on Brunauer–Emmett–Teller (BET) analysis, the specific surface area, total pore volume, and average pore radius were determined to be $8.95 \text{ m}^2 \text{ g}^{-1}$, $2.77 \times 10^{-2} \text{ cm}^3 \text{ g}^{-1}$, and 6.19 nm , respectively. The relatively low surface area further supports the absence of significant microporous structure and is consistent with the Type III isotherm behavior.

3.3 Optical Properties

The optical properties of the synthesized particles were investigated using UV–Vis diffuse reflectance spectroscopy (UV–Vis DRS) and photoluminescence (PL) spectroscopy. **Fig. 4a** shows the UV–Vis DRS spectrum of the synthesized ZnO sample. A strong absorption edge is observed at approximately 390 nm, which corresponds to the electronic transition from the valence band to the conduction band of ZnO. In addition, a noticeable absorption in the visible-light region is observed. This feature may be associated with the presence of defect states within the ZnO lattice, such as oxygen vacancies or zinc interstitials, which can introduce intermediate energy levels and lead to bandgap narrowing (3.02 eV).

The contribution of residual carbonaceous species derived from the plant extract cannot be entirely excluded. However, if present, their amount is likely minimal and may fall below the detection limit of Raman spectroscopy. In the absence of direct characterization (e.g., XPS), the presence and role of such species cannot be conclusively established. Therefore, the observed visible-light absorption is attributed to a combination of intrinsic defect states and possible extrinsic contributions, which cannot be independently distinguished in the present study.

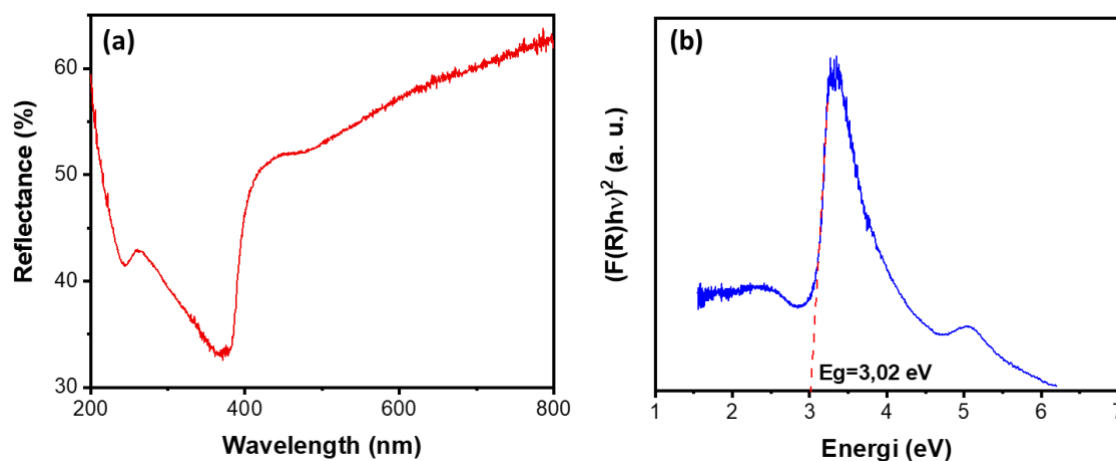
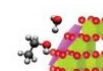


Figure 4. (a) Reflectance spectra of synthesized particles; (b) Tauc plot for direct bandgap to determine the bandgap energy.



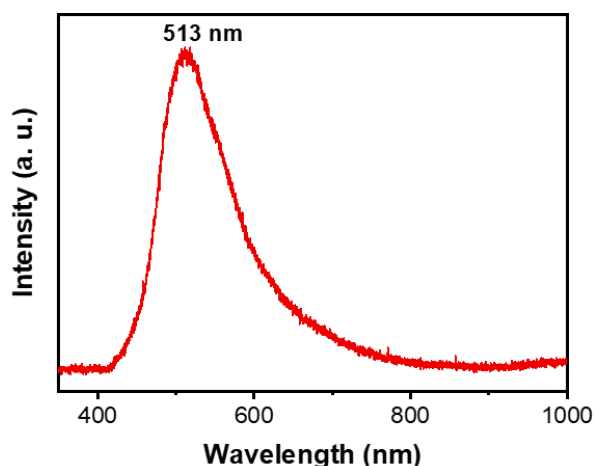


Figure 5. PL spectra of the synthesized ZnO.

Further investigation, including comparative analysis with a control ZnO sample, would be required to clarify the origin of this absorption.

ZnO is a direct-bandgap semiconductor. Its optical bandgap energy was estimated using the Kubelka–Munk equation for direct bandgap. **Fig. 4b** presents the Tauc plot derived from the Kubelka–Munk function of the synthesized ZnO sample. The linear portion of the plot was extrapolated to the energy axis, and the intercept corresponds to the direct bandgap energy of the material. Based on this analysis, the bandgap energy of the synthesized ZnO was determined to be 3.02 eV.

The luminescence behavior and defect-related electronic states of the synthesized particles were further examined by room-temperature photoluminescence spectroscopy. **Fig. 5** displays the PL emission spectrum of the synthesized ZnO. A dominant emission peak is observed at approximately 513 nm, corresponding to an emission energy of 2.41 eV. This emission lies in the green to yellow-green region of the visible spectrum, although contributions from other visible emissions are also present. The main emission centered at 513 nm originates from radiative recombination processes involving defect-related energy levels within the ZnO bandgap. These defect states facilitate electronic transitions through intermediate levels, resulting in visible-light emission.

Visible-range PL emissions in ZnO are commonly associated with intrinsic point defects, such as zinc interstitials (Zn_i), oxygen vacancies (V_O), zinc vacancies (V_{Zn}), and oxygen interstitials (O_i). Theoretically, the energy levels of Zn_i , O_i , and V_{Zn} are located at approximately 0.22, 2.28, and 3.06 eV below the conduction band, respectively, while the V_O level is situated about

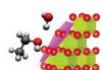
0.9 eV above the valence band. Green emission is generally attributed to electron transitions from the conduction band to V_O states, whereas yellow emission arises from transitions involving Zn_i and V_O defect levels [20,21]. The presence of these defect states is typical for ZnO synthesized via green synthesis routes, where organic precursors and mild reaction conditions strongly influence defect formation and optical behavior [10].

The observed defect-related emission in the photoluminescence spectra is interpreted with consideration that defect density is not solely governed by crystallite size. While smaller crystallite sizes are often associated with increased defect concentrations due to higher surface-to-volume ratios, such a direct correlation is not assumed in this study. Instead, the presence of prominent defect-related emission is discussed as being more strongly influenced by synthesis conditions, including the use of bio-derived species, possible non-equilibrium growth processes, and structural disorder within the ZnO lattice. These factors may promote the formation of intrinsic defects such as oxygen vacancies and zinc interstitials, which contribute to the observed visible emission. Therefore, the defect-related optical behavior is considered to arise from the combined effects of synthesis-induced disorder and surface states, rather than crystallite size alone [22–24].

3.4 Photocatalytic Activity

The photocatalytic activity of the synthesized ZnO particles was evaluated to assess their ability to remove Rhodamine B (RhB) under irradiation. Prior to light exposure, the suspension was kept in the dark to establish adsorption–desorption equilibrium. However, the extent of RhB adsorption was not quantitatively determined in this study. During the photocatalytic process, aliquots of the solution were collected at 30 min intervals and analyzed by UV–Vis spectroscopy. **Fig. 6a** shows the temporal evolution of the UV–Vis absorption spectra of RhB in the presence of the synthesized ZnO. A gradual decrease in the maximum absorption intensity of RhB is observed with increasing irradiation time, indicating effective removal of the dye molecules.

The absorbance data were subsequently converted into concentration values, which were used to calculate the overall removal efficiency. **Fig. 6b** presents the evolution of RhB removal efficiency as a function of irradiation time, reaching a maximum value of 97.68% after 150 min. It should be noted that this value reflects the



combined contribution of adsorption and photocatalytic degradation.

The photocatalytic degradation kinetics of RhB were analyzed using a pseudo-first-order reaction model. **Fig. 6c** shows the corresponding kinetic plot, which exhibits a reasonable linear fit ($R^2 = 0.9795$). The apparent rate constant (k) was determined to be 0.0290 min^{-1} from the slope of the linear regression. A slight deviation from

linearity is observed at 150 min, which may be attributed to the system approaching low concentration limits, where the pseudo-first-order assumption becomes less valid. Additionally, factors such as intermediate formation, changes in surface activity, or partial deactivation of active sites at prolonged irradiation times may also contribute to this deviation.

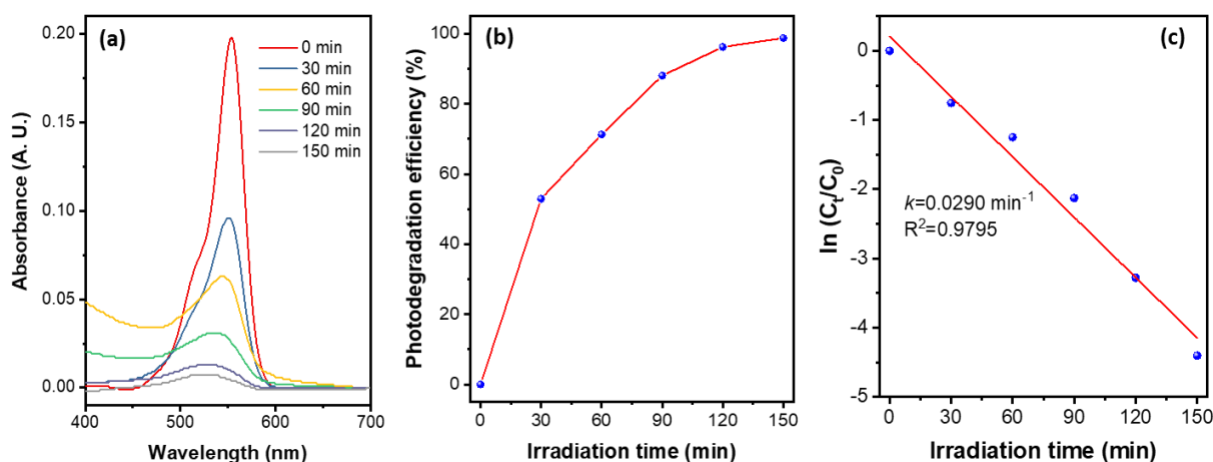


Figure 6. Photocatalytic activity of synthesized ZnO; (a) absorption spectra of Rhodamine B during photodegradation, (b) photodegradation efficiency; (c) pseudo first order kinetic plot.

The observed photocatalytic performance may be associated with the structural and optical characteristics of the synthesized ZnO, including crystallite features, defect-related states, and visible-light absorption. However, the specific contributions of these factors, as well as the relative roles of adsorption and photocatalytic degradation, cannot be conclusively distinguished in the absence of a control sample and quantitative adsorption analysis.

The photocatalytic mechanism is proposed based on established ZnO pathways, where photogenerated electrons and holes may produce reactive species such as $\cdot\text{O}_2^-$ and $\cdot\text{OH}$ that contribute to Rhodamine B degradation [3,6]. However, the specific roles of these species cannot be conclusively determined, as no radical scavenging, ESR, or charge separation analyses were performed in this study.

4 Conclusion

ZnO particles were successfully synthesized via a green route using shallot peel extract, and their structure–property relationships were systematically evaluated. The results confirmed the formation of phase-pure wurtzite ZnO with rod-like morphologies composed of aggregated crystallites. Optical analysis revealed a bandgap of

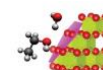
3.02 eV and defect-related visible-light absorption. The synthesized ZnO showed high photocatalytic performance toward Rhodamine B degradation, achieving 97.68% removal within 150 min following pseudo-first-order kinetics. However, in the absence of a control ZnO sample, the specific role of the extract in influencing crystallite size, defect formation, and photocatalytic activity cannot be conclusively established. In addition, the contribution of adsorption and the exact origin of visible-light absorption remain to be further clarified. Future work should include comparative studies and additional characterization to address these aspects. Overall, this study highlights the correlation between synthesis conditions, structural features, and photocatalytic behavior in green-synthesized ZnO systems.

Acknowledgement

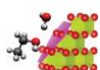
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