

Article

Effect of Calcination Temperature on the Photocatalytic Activity of Precipitated ZnO Nanoparticles for the Degradation of Rhodamine B Under Different Light Sources

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Academic Editor: Chengyun Zhou

Received: 2 November 2024

Revised: 23 December 2024

Accepted: 23 December 2024

Published: 26 December 2024

Citation: Saidani, A.; Boudraa, R.; Fendi, K.; Benouadah, L.; Benabbas, A.; Djermoune, A.; Salvestrini, S.; Bollinger, J.-C.; Alayyaf, A.A.; Mouni, L. Effect of Calcination Temperature on the Photocatalytic Activity of Precipitated ZnO Nanoparticles for the Degradation of Rhodamine B Under Different Light Sources. *Water* **2025**, *17*, 32. <https://doi.org/10.3390/w17010032>

Correction Statement: This article has been republished with a minor change. The change does not affect the scientific content of the article and further details are available within the backmatter of the website version of this article.

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Abstract: This research provides valuable insights into the application of ZnO nanoparticles in photocatalytic wastewater treatment. Process optimization was carried out by determining the ratio of the surface area to the energy band gap (S/E) in the photocatalysis rate under different sources of light (UV light, visible light, sunlight). The nanoparticles were synthesized using the precipitation technique, and the calcination process was carried out within a temperature range of 400 to 700 °C. The structural, morphological, and optical properties of materials were investigated using X-ray powder diffraction (XRD), scanning electron microscopy (SEM), UV-Vis diffuse reflectance (UV-Vis DRS), Raman spectroscopies, and Fourier transform infrared (FTIR) spectroscopies. The study demonstrates that calcination temperature significantly influences the photocatalytic activity of ZnO nanoparticles by altering their size, surface properties, shape, and optical behavior. Optimal decomposition efficiencies of Rhodamine B were achieved at 400 °C, with yields of 24%, 92%, and 91% under visible, UV, and sunlight irradiation, respectively. Additionally, the surface area decreased from 12.556 to 8.445 m²/g, the band gap narrowed slightly from 3.153 to 3.125 eV, and crystal growth increased from 0.223 to 0.506 μm as the calcination temperature rose. The photocatalytic properties of ZnO nanoparticles were assessed to determine their efficiency in decomposing Rhodamine B dye under operational parameters, including pollutant concentration (C₀), sample amount, pH level, and reaction time. The sample exhibited the best breakdown rates with C₀ = 5 mg/L, solid-to-liquid ratio (S/L) = 50 mg/L, pH = 7, and reaction time = 1 h. Additionally, we combined two oxidation processes, namely H₂O₂ and photocatalytic oxidation processes, which significantly improved the Rhodamine B removal efficiency, where 100% of RhB was degraded after 60 min and 100 μL of H₂O₂.

Keywords: nanoparticles; photocatalytic activity; rhodamine B; sunlight; UV light; visible light; ZnO

1. Introduction

Solar energy is one of the most important renewable energy sources, if not the most important, in terms of availability, abundance, and affordability [1–3]. Researchers are exploring photocatalysis as a transformative technology to address environmental challenges. Photocatalysis harnesses sunlight, a renewable energy source in water treatment, by effectively breaking down harmful pollutants like dyes into less toxic substances, reducing their environmental and health risks and minimizing their impact on ecosystems. This eco-friendly solution contributes to cleaner air, water, and soil. It aligns with global efforts toward greener, more energy-efficient technologies [4–7]. Moreover, photocatalysis enables the direct transformation of solar energy into usable heat or other valuable energy resources, sparking significant interest and applications across various sectors. Concretely, photocatalytic technologies have found utility in sensitized solar cells [8,9], chemical pollutant oxidation, and reduction [10], as well as the inactivation of microbial pathogens. Experts claim that photocatalysis is a complex process that fundamentally depends on the presence of a catalyst, usually a semiconductor. This process influences the kinetic rate of the photochemical reaction involving reactive species and light, while maintaining its fundamental thermodynamic limitations. Photons serve as the energy source, aligning with the band gap energy of the semiconductor, to excite electrons from the valence band to the conduction band, resulting in the generation of electron–hole pairs. Charge carriers migrate to the photocatalyst surface, where they participate in redox reactions that generate reactive oxygen species (ROS), including hydroxyl radicals ($\bullet\text{OH}$) and superoxide anions (O_2^-), essential for pollutant degradation [11–13]. Zinc oxide (ZnO) is a widely recognized, and highly attractive semiconductor, valued for its remarkable properties such as high crystallinity, tunable optical and electronic properties, exceptional quantum efficiency, low electrical conductivity, and excellent thermal stability [14,15]. These properties can be tailored by synthesizing ZnO in various nanostructural forms, such as nanoparticles, nanorods, nanosheets, and hierarchical architectures [16], each offering distinct advantages for diverse applications like anti-corrosion [17], electronics and optoelectronics [18], industrial applications [19], nanomedicine applications like cancer therapy [20], antioxidant [21] and antibacterial uses [22], and environmental protection like photocatalysis for pollutant degradation [23,24]. Zinc oxide ZnO is often considered superior to titanium dioxide TiO_2 for sunlight photocatalysis [9,25,26].

Zinc oxide is classified as an n-type semiconductor owing to the presence of extra zinc atoms and oxygen vacancies in the interatomic sites. Its crystalline structure adopts the hexagonal wurtzite form [27,28]. It is recognized that its activity in the ultraviolet region is attributed to its broad band gap of 3.3 eV. However, as the band gap energy decreases, the energy threshold for electronic transitions is lowered, allowing the absorption of lower energy photons and shifting the material's radiation absorption towards the visible spectrum [29,30]. This transition is essential for improving the performance of ZnO in sunlight [31], an objective actively targeted by researchers in the field by exploring several strategies, including doping with metal or non-metal elements, creating heterojunctions with other materials, and modifying defect density to optimize light absorption and charge carrier dynamics [32]. Another crucial consideration is the assessment of the suitability of a material as a catalyst by subjecting it to specific conditions for gaining a higher specific surface area and a larger number of active sites to achieve more efficiency with less quantity

because we should mention that the big quantity of using ZnO pose ecological risks to aquatic and terrestrial life, affecting their health, disrupting ecological processes, and altering ecosystem balance. They accumulate in aquatic organisms, disrupt soil microbiota, and pose risks to non-target organisms. Green synthesis of biocompatible ZnO NPs offers potential for environmental remediation, but comprehensive ecotoxicological assessments are needed for safety, as well as improved thermal and mechanical stability.

It is important to recognize that the performance of the material can be tuned by adjusting its particle size and morphology [33]. The surface characteristics of metal oxides, including oxygen vacancies and surface defects, are essential in improving photocatalytic activity by offering active sites for reactions. As the calcination temperature rises, particle aggregation diminishes the surface area and defect density, resulting in a reduction of active sites. This process enhances crystallinity while marginally increasing the band gap, resulting in a shift of light absorption towards the UV region and restricting the utilization of visible light. Optimizing photocatalytic performance necessitates a balance among surface area, defect density, and band gap energy [34–37]. It is important to find a compromise between band gap energy and surface area where a catalyst exhibits its optimal photocatalytic performance. Therefore, achieving an efficient photocatalyst requires implementing two contradictory conditions while controlling the thermal treatment. Extensive research has been conducted on the effects of thermal treatments on ZnO on properties such as optical properties [34,36], structural morphology [38,39], and photonic efficiency [33,40]. However, there is a void in the understanding of the inverse relationship between band gap, area, and photonic energy. This study provides a unique perspective. By evaluating how this ratio affects photonic efficiency across different light sources, the work bridges the gap between material characterization and performance optimization. The focus of our study is a comprehensive comparison of the effect of calcination temperature on the photonic performance of ZnO nanoparticles under different light sources UV, visible, and solar radiation. This approach allows us to determine the optimal calcination conditions for specific light sources. This research is based on the fabrication of ZnO nanostructures using the deposition method with the calcination temperature varying from 400 °C to 700 °C. Zinc oxide (ZnO) nanoparticles can be synthesized using various methods, including sol–gel [41], hydrothermal [42], chemical vapor deposition [43], microwave-assisted [44], solution combustion [45], and co-precipitation [38]. Each method offers unique advantages and applications, with the choice depending on the specific application requirements and desired properties. Precipitation is considered one of the most efficient processes for producing ZnO nanoparticles. The preference for this method stems from its straightforward synthesis, low formation temperature, high level of sample homogeneity, and the ability to precisely control the chemical composition [46,47]. The primary goal is to achieve the optimal calcination temperature, ensuring the highest possible photocurrent yield under UV, visible light, and sunlight. The secondary goal is to determine a favorable ratio between small band gap energy and large specific surface area, resulting in high efficiency in the degradation of the organic pollutant [44]. After determining the optimal light source and calcination temperature, the catalytic process is optimized by adjusting both the ZnO mass and the pollutant concentration, along with pH adjustments. A complementary investigation is also conducted on the effect of adding H₂O₂ to the photocatalytic system to achieve maximum efficiency. Rhodamine B was chosen as a model pollutant of choice to evaluate the photocatalytic performance of the synthesized samples. This dye is often used in various industrial applications and has been observed to pose potential environmental hazards. Trace experiments have indicated its potential ecotoxicity towards freshwater aquatic organisms [48,49]. Lethal doses of this substance have the potential to cause toxicity,

and its introduction into the natural ecosystem may pose risks to aquatic organisms, as well as carcinogenic effects on both humans and animals [50].

2. Materials and Methods

The materials used in this study included zinc chloride (anhydrous) (ZnCl_2 , >97%, MW = 81.89 g/mol, Sigma-Aldrich, Germany), sodium hydroxide (NaOH, 99%, Merck, Germany), and Rhodamine B dye (RhB) ($\text{C}_{28}\text{H}_{31}\text{ClN}_2\text{O}_3$, purity 95%, Biochem, France). All materials were used without any additional purification steps. Deionized water (DI) was obtained from local sources and used in the experimental procedures.

2.1. Synthesis of ZnO Nanopowders

ZnO was prepared by precipitation with a key modification in the addition of a saturated NaOH solution all at once rapidly instead of gradual addition [51] involving the following steps. At room temperature (26 °C), 30 g of zinc chloride (ZnCl_2) was dissolved in 100 mL of deionized water (DI) to form a solution. The solution underwent continuous agitation using a stirrer plate at room temperature, and upon achieving homogeneity, 15 mL of a sodium hydroxide-saturated solution (NaOH) was added at once. The mixture was vigorously stirred for 15 min at room temperature, resulting in the formation of a white precipitate. This precipitate was filtered by filter paper with a pore size equal to 0.45 μm and meticulously rinsed with DI to eliminate all salt and chloride residues. The obtained zinc hydroxide was then dried at 100 °C and divided into five parts. Each part was then treated in air at a specific temperature in the range of 400 °C to 700 °C for 2 h starting from room temperature with a heating rate of 5 °C/min. The synthesized samples were designated as follows: ZnO-400, ZnO-500, ZnO-600, and ZnO-700, corresponding to the calcination temperature.

2.2. Characterization

Powder X-ray diffraction (XRD) patterns were recorded in the range of 20–80° (2 θ) in the scan mode (0.028 step size, 2 s counting time) using an Empyrean Panalytical (Germany) powder diffractometer employing $\text{CuK}\alpha$ radiation operating at 40 kV and 40 mA. The unit cell parameters were refined within the hexagonal system through the measured peak positions of the XRD pattern, with a least squares method and employing the CELREF program. For the determination of peak characteristics, the peak profiles were fitted with the WINPLOTR program. The crystallite sizes and microstrain contributions were estimated from analyses of Williamson–Hall plots [52]. For morphological studies, a scanning electron microscope (SEM) of the Thermo Scientific Quattro S (USA) field emission type was utilized. The functional groups in samples were identified using a Fourier-transform infrared spectrometer (FT-IR), specifically the Thermo Scientific Nicolet IS50 FT-IR spectrometer (USA), covering the wavenumber range of 400–4000 cm^{-1} . Raman spectroscopy was performed using an Avantes (AvaSpec-HSC-TEC spectrometer, Apeldoorn, The Netherlands), ensuring precise detection of Raman signals over the spectral range of 200–4000 cm^{-1} . Additionally, optical absorbance in the range of 200–800 nm was measured using an Evolution 220 (Thermo Scientific, Waltham, MA, USA) UV-visible spectrophotometer. Surface area measurements were conducted at 77 K using an Autosorb iQ Station Analyzer (Quantachrome, Boynton Beach, FL, USA), providing detailed insights into the surface properties of the samples.

2.3. Photocatalytic Experiments

Rhodamine B (RhB) served as the model dye to evaluate the photocatalytic performance of the various samples of ZnO. The photodegradation tests were conducted under distinct irradiation sources, including solar (1800 Lux), visible (using 150 W LED lamps),

and UV light (365 nm, rated at 15 W). The tests were performed in beakers containing 50 mg of photocatalyst and 100 mL of an aqueous solution of RhB dye (5 mg/L). Prior to the photocatalytic process, the solutions underwent magnetic stirring for 120 min in the dark to assess the absorbance yield of sample even the adsorption equilibrium was reached within 30 min. Subsequently, the mixture was exposed to different light irradiation sources to evaluate the impact of heat treatment conditions on the photocatalytic efficiency.

The influence of pH on photocatalytic degradation was examined by modifying the pH of a 5 mg/L Rhodamine B solution with 0.1 M NaOH and 0.1 M HCl, measuring pH values (2, 7, 10) using a calibrated pH meter. For H₂O₂, 100, 300, and 500 microliters were added to the reaction mixture while keeping the catalyst dosage and reaction volume constant. Kinetic studies were conducted by collecting samples at 30-min intervals for those irradiated by visible and UV light, and at 15-min intervals for samples exposed to sunlight. For the effects of H₂O₂ and pH, the interval was 5 min. The samples were centrifuged to remove the catalyst, and the supernatant was analyzed using UV-Vis spectrophotometry.

The photodegradation yield (D (%)) was estimated using:

$$D(\%) = \frac{(C - C_t)}{C} \times 100 \quad (1)$$

where C and C_t represent the RhB concentration after adsorption equilibrium on the semiconductor before irradiation and at a given time, respectively. The photodegradation kinetics of RhB were evaluated using the Langmuir–Hinshelwood equation [53] (Equation (2))

$$r = -\frac{dC_t}{dt} = \frac{bKC_t}{1 + KC_t} \quad (2)$$

In photocatalysis experiments, the concentrations used are generally low, so the integrated Langmuir–Hinshelwood equation can be simplified as follows:

$$C_t = C \times e^{-kt} \quad (3)$$

where b (mg·min^{−1}·L^{−1}), K (L·mg^{−1}), and k (= b × K, min^{−1}) are the photocatalytic constant, the adsorption constant and the first-order reaction rate constant, respectively.

3. Results

3.1. Material Characterization

3.1.1. X-Ray Powder Diffraction

The X-ray diffraction patterns are shown in Figure 1. ZnO is shown as a single pure phase. Refinement of the lattice parameters for this phase as a function of the calcination temperature shows no important differences (Table 1); the observed values are in accordance with those of JCPDS reference. Their very slight decrease upon rising calcination temperature is associated with the increase of crystal sizes. In this respect, analysis by means of Williamson–Hall method of the evolution of crystallite size and microstrain confirms this tendency. As expected, the crystallite size D and microstrain ε (Δd/d%) are found to vary in opposite directions vs. calcination temperature (Table 2). Both results reveal the decrease in the number of defects and the enhancement of crystallinity while calcination temperature increases, as corroborated by other research. This observation will have a profound impact on the optical properties. It is important to note that the grain sizes in a polycrystalline sample is independent of the crystallite sizes [54], although in some cases these two quantities are correlated. The former is deduced from electron microscope observations while the latter are estimated from X-ray diffraction peak profiles.

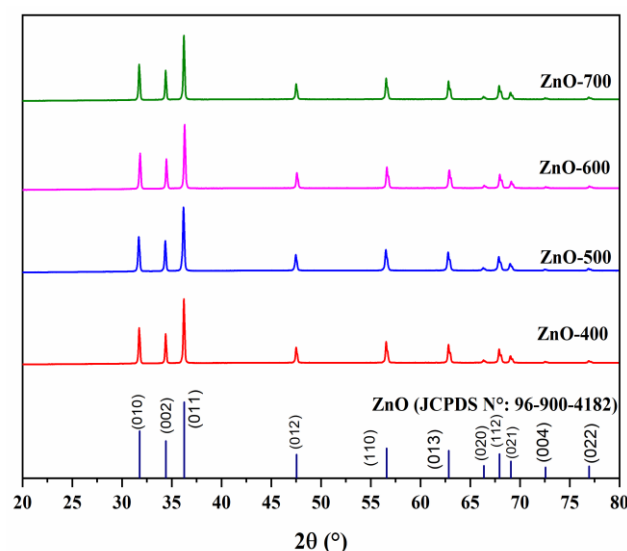


Figure 1. XRD spectra of ZnO calcined at 400 °C, 500 °C, 600 °C, and 700 °C.

Table 1. Lattice parameters, crystallite size D , and microstrain ε of ZnO phase in the four samples.

Calcination Temperature (°C)	Crystallite Size D (nm)	Crystal Parameters (Å)		Microstrain ε (%)
		A	C	
ZnO-400	63	3.2502(1)	5.2068(2)	0.0335
ZnO-500	66	3.2498(1)	5.2062(3)	0.0177
ZnO-600	67	3.2497(1)	5.2056(2)	0.0139
ZnO-700	72	3.2498(1)	5.2058(2)	0.0061

Table 2. BET analysis results of synthesized ZnO samples.

Samples	BET Surface Area (m ² /g)	Langmuir Surface Area (m ² /g)	BJH Adsorption Average Pore Diameter (nm)	BJH Desorption Average Pore Diameter (nm)	Pore Volume (cm ³ /g)
ZnO-400	2.793	12.556	27.238	26.453	0.0048
ZnO-500	2.640	10.733	32.039	25.904	0.0054
ZnO-600	2.518	9.126	32.268	22.965	0.0036
ZnO-700	2.201	8.445	15.467	9.518	0.0026

3.1.2. Scanning Electron Microscopy (SEM)

In this investigation, we consequently explored the impact of diverse calcination temperatures, ranging from 400 °C to 700 °C, on the morphology and size of zinc oxide (ZnO) nanoparticles (Figure 2). At 400 °C, the nanoparticles homogeneously exhibited roughly spherical shapes. At intermediate calcination temperatures, an evolution towards larger agglomerations and well-faced crystals is evident, reflecting enhanced sintering and crystal growth from 0.112 to 0.506 μm . Parallely, the spread of the particle size distribution is more extended. At 700 °C, these tendencies are more pronounced. These results correspond to those presented in a similar study [55]. This comprehensive approach allowed us to discern nuanced changes in morphology and particle size at different temperature regimes, providing valuable insights for tailoring the synthesis of ZnO nanoparticles to specific applications, whether requiring finer particles at lower temperatures or larger, faceted structures at higher temperatures. Additionally, our findings shed light on how these temperature variations impact the optical properties and crystal structure of ZnO, enriching our understanding of the multifaceted implications for various applications in catalysis and

photocatalysis. The reduced sizes and higher defect levels at lower temperatures suggest higher specific surface area and density of active sites. The correlation between the nuanced morphological changes induced by varying calcination temperatures and the subsequent effects on catalytic and photocatalytic activities provides a comprehensive framework for optimizing the synthesis of ZnO nanoparticles for specific functional applications.

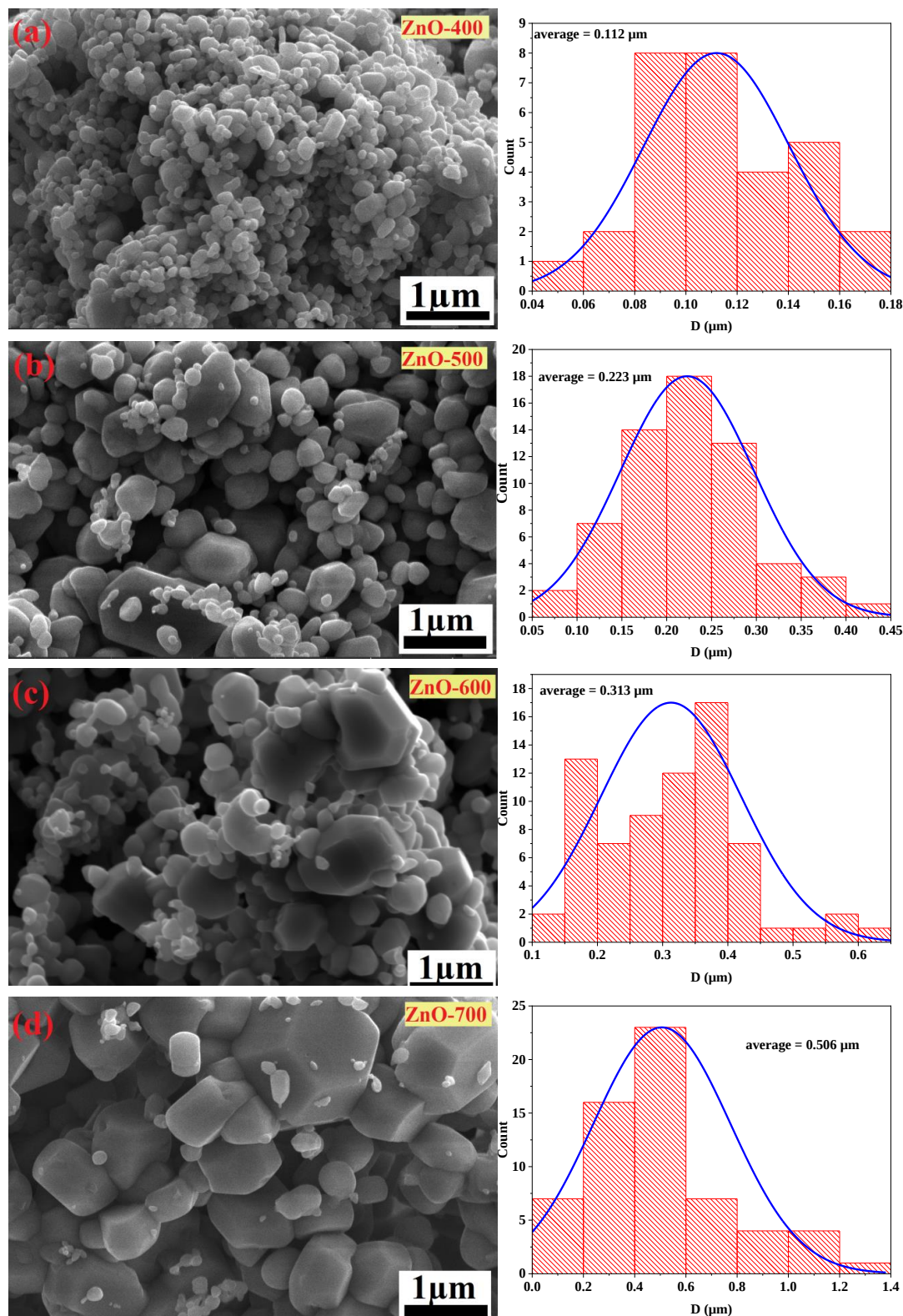


Figure 2. SEM image for different samples of ZnO nanoparticles calcined at 400 °C, 500 °C, 600 °C, and 700 °C.

3.1.3. Fourier Transform Infrared (FTIR)

Figure 3 shows the FTIR spectra of the samples synthesized under different heat treatment conditions for ZnO nanoparticles. In the spectral range between 1600 and 1700 cm^{-1} , the zinc oxide peak became sharper and smoother, indicating increased crystallinity as the calcination temperature increased. A slight peak shift was observed, specifically between 435.06 and 459.16 cm^{-1} . Furthermore, in the calcined samples above 400 $^{\circ}\text{C}$, the disappearance of the OH band at 3492.70 cm^{-1} suggests the achievement of the transformation from $\text{Zn}(\text{OH})_2$ to ZnO [56]. During the first step of preparation, the presence of excess OH^- ions led to the formation of $\text{Zn}(\text{OH})_2$, which further transformed into ZnO during the calcination process. These findings support the results obtained from XRD analysis confirming the transformation of $\text{Zn}(\text{OH})_2$ into ZnO and enhancing the overall reliability of the experimental observations. The examination of the FTIR and XRD results shows that chemical changes in the ZnO nanoparticles occur during calcination.

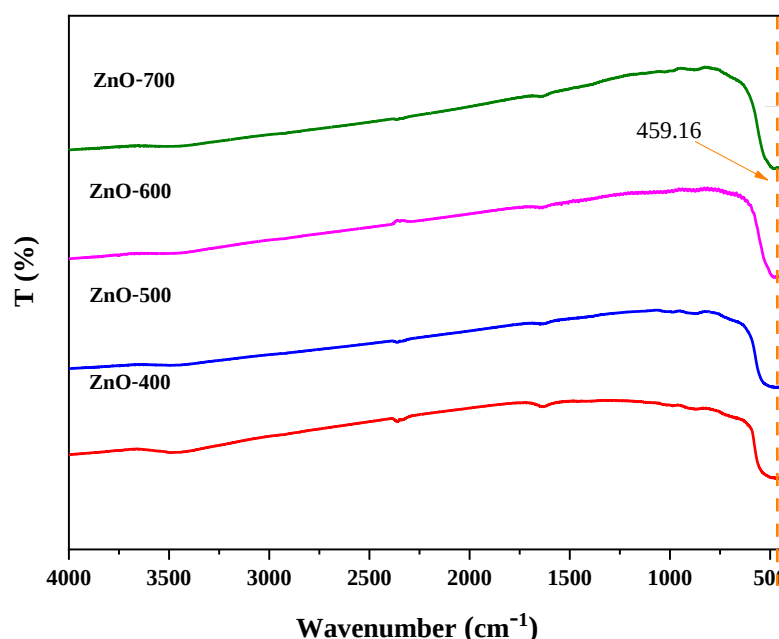


Figure 3. FTIR spectra of ZnO nanoparticles calcined at 400 $^{\circ}\text{C}$, 500 $^{\circ}\text{C}$, 600 $^{\circ}\text{C}$, and 700 $^{\circ}\text{C}$.

3.1.4. BET Surface Area

The BET analysis of the four ZnO samples, subjected to different calcination temperatures (400 $^{\circ}\text{C}$, 500 $^{\circ}\text{C}$, 600 $^{\circ}\text{C}$, 700 $^{\circ}\text{C}$), revealed distinctive patterns in their surface properties. Figure 4 shows the N_2 adsorption–desorption isotherms for both the ZnO precursor and the samples treated at different temperatures. The observations indicated a continuous decrease in the specific surface area from 12.556 to 8.445 m^2/g with the increase in calcination temperature from 400 $^{\circ}\text{C}$ to 700 $^{\circ}\text{C}$, as detailed in Table 2. This decrease indicates a reduction in the available surface area, resulting from the fusion and agglomeration of particles at high temperatures, as clearly shown by scanning electron microscope images that highlight the increase in the size of crystalline particles.

When analyzing the pore size distribution, it was observed that the samples exposed to high calcination temperatures showed a significant shift towards larger pores, reflecting a relative increase in larger pores at the expense of smaller ones. However, the analysis showed a decrease in the total pore volume with increasing calcination temperature, due to particle coalescence and the shrinkage of small pores.

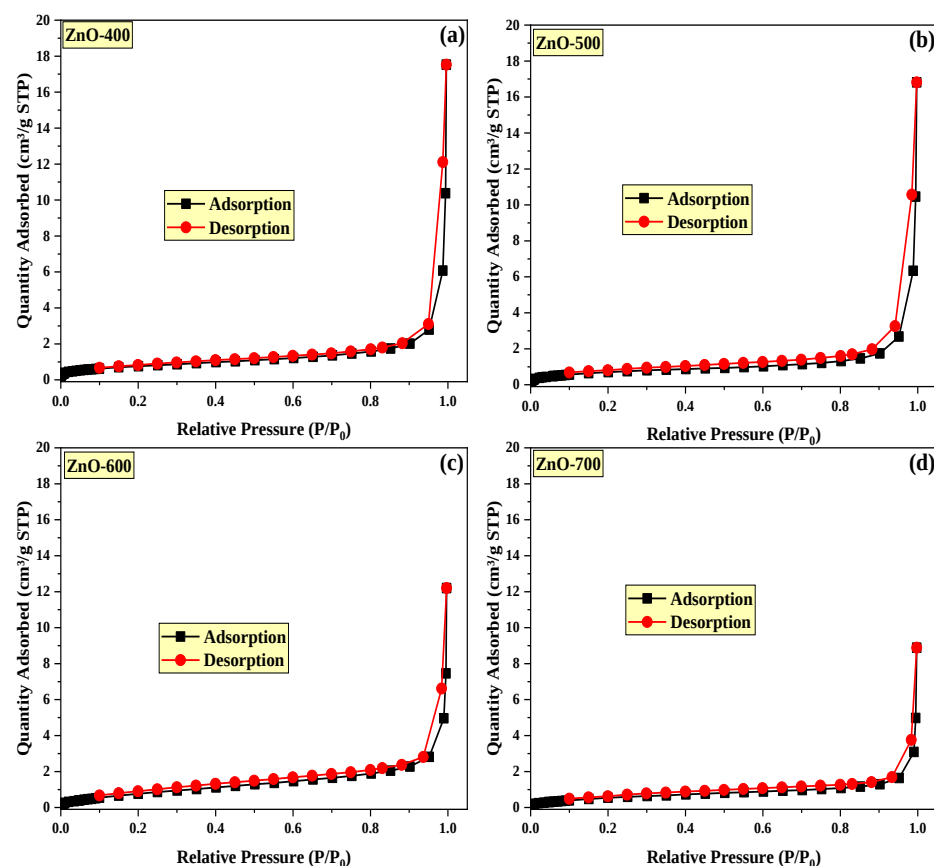


Figure 4. Nitrogen adsorption–desorption isotherms for synthesized ZnO samples calcined at (a) 400 °C, (b) 500 °C, (c) 600 °C, and (d) at 700 °C.

Similar to the current results, another study indicated similar effects on different materials. For example, a study showed a significant decrease in surface area from 25.4 to 8.8 m²/g and pore volume from 0.35 to 0.03 cm³/g with an increase in temperature up to 600 degrees Celsius [57]. Furthermore, a study on zirconia gel showed a significant decrease in surface area from 142 to 93 m²/g when calcined up to 500 degrees Celsius, with the complete elimination of micropores at 700 degrees Celsius [58]. This indicates a reduction in the smaller pores, along with a significant increase in crystallization.

3.1.5. Raman Spectroscopy

The Raman spectrum of ZnO nanoparticles calcined at 400 °C, 500 °C, 600 °C, and 700 °C demonstrates the progression of crystal quality, defect concentration, and structural characteristics as the temperature rises. The notable E₂ (high) mode (~437 cm^{−1}), illustrated in Figure 5, is indicative of the wurtzite ZnO lattice structure [59], which sharpens and intensifies at 600 °C, indicating improved crystallinity and well-ordered lattice vibrations. At lower temperatures (400 °C, 500 °C), broader peaks, and higher defect-related signals [60], the A₁(LO) mode, approximately at 600 cm^{−1}, is detected, indicating reduced crystallite dimensions and the existence of oxygen vacancies, along with lattice imperfections. At 700 °C, the high level of crystallinity is maintained; however, the minor reduction in peak intensity indicates that grain growth is occurring alongside a decrease in surface scattering effects, potentially leading to a weaker Raman signal. These trends underscore the interaction among defect annealing, grain size, and lattice ordering [61].

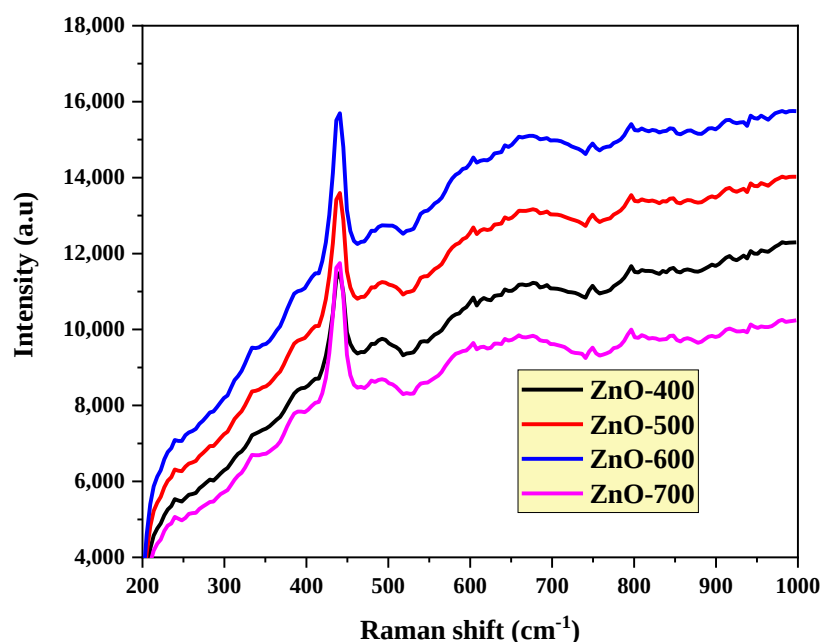


Figure 5. Raman spectrum of ZnO nanoparticles calcined at 400 °C, 500 °C, 600 °C, and 700 °C.

3.1.6. Optical Properties

The optical absorption characteristics of ZnO nanoparticles were explored using UV-Vis spectroscopy within the wavelength range of 200 to 800 nm (Figure 6). Evidently, ZnO, being a high band gap semiconductor, showcases distinctive properties in the visible and near-ultraviolet spectra. The determination of the optical band gap of the nanoparticles was calculated using the Tauc formula which analyzes the linear portion of the graph between the square of the absorption coefficient ($\alpha h\nu$) and photon energy ($h\nu$) [62]:

$$(\alpha h\nu)^n = B(h\nu - E_g) \quad (4)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, E_g is the optical band gap energy in eV, B is a proportionality constant, n is the exponent depending on the electronic transition type, $n = 1/2$ for directly allowed transitions, and $n = 2$ for indirectly allowed transitions [63]. The optical band gap of the ZnO nanoparticles was found to be temperature-dependent, exhibiting a decrease in band gap energy as the calcination temperature increased. The absorption maxima of the nanoparticles, calcined at various temperatures (400 °C, 500 °C, 600 °C, and 700 °C), shifted towards lower wavelengths, specifically at 371, 373, 375, and 376 nm Figure 7, respectively. Correspondingly, the band gaps were measured as 3.153, 3.139, 3.128, and 3.125 eV, respectively, as presented in Table 3 and Figure 6. These findings are consistent with various similar studies [33,63]. The observed blue shift in the band edge absorption wavelength of ZnO nanopowders produced at high temperatures is generally attributed to the presence of shallow levels within the band gap, which can be linked to impurity atoms or disruptions in the ZnO lattice [37,64,65]. However, it is suggested that this red shift is mainly due to the decrease in band gap resulting from an increase in particle size at higher calcination temperatures. This increase in particle size leads to a reduction in specific surface area, causing an increase in the coordination between surface atoms and a decrease in the coordination between surface atoms and underlying atoms. This, in turn, contributes to the observed blue shift phenomenon, attributed to the reduction in interatomic distance in the surface.

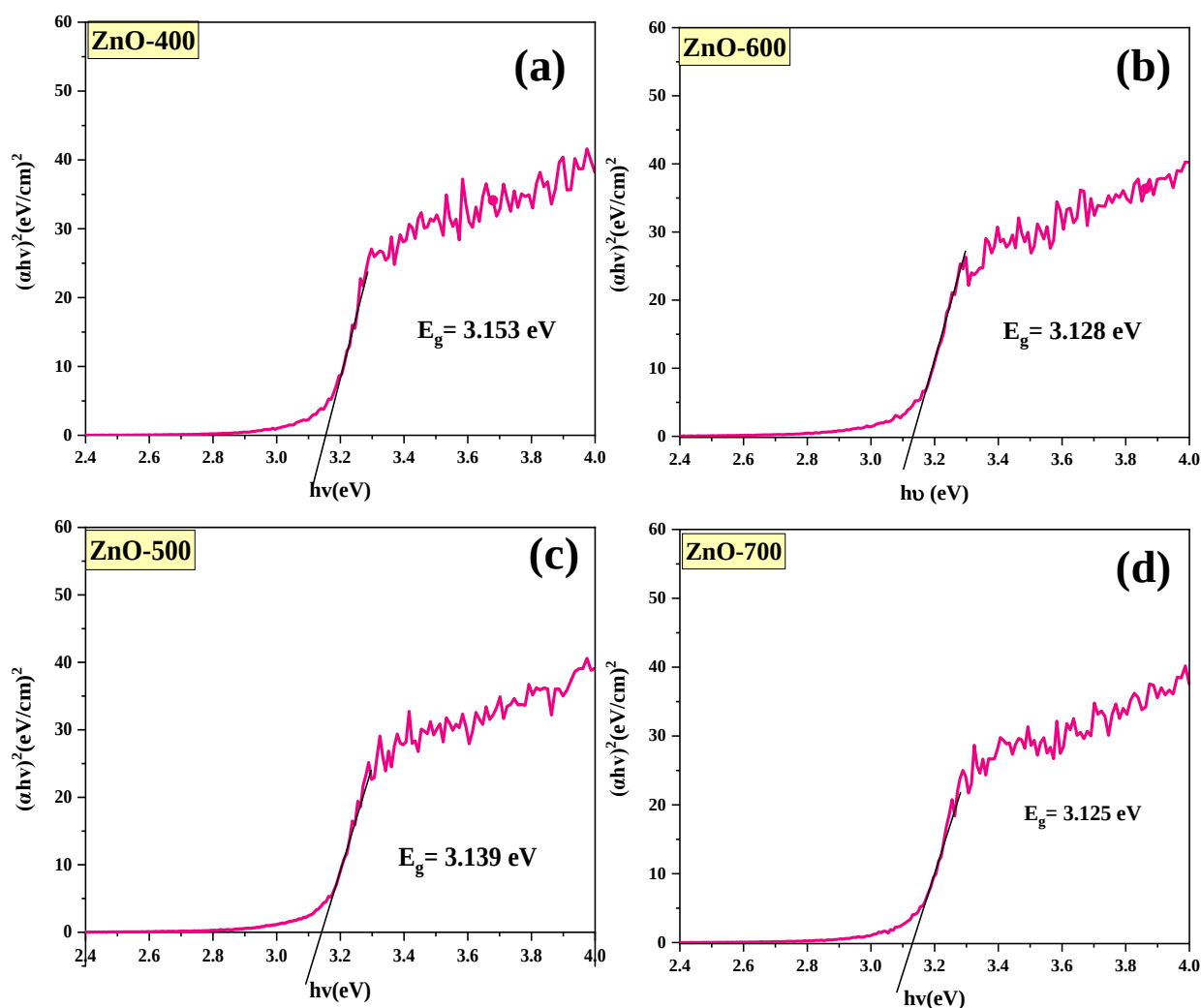


Figure 6. The optical properties and Tauc plots for determining the band gap energy of the photocatalyst calcined at 400 °C, 500 °C, 600 °C, and 700 °C.

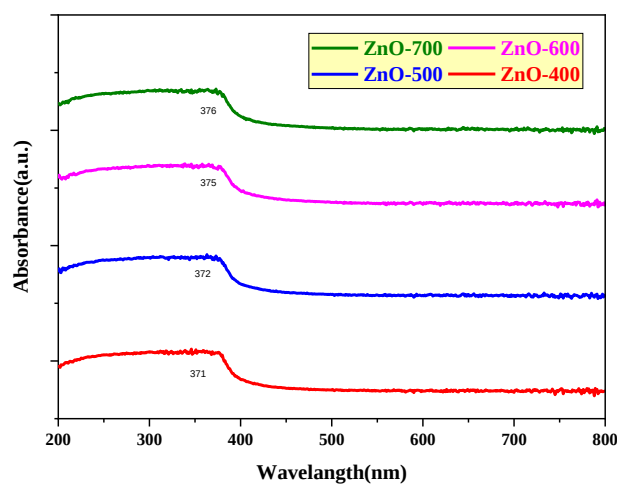


Figure 7. UV-visible absorption spectra of ZnO nanoparticles calcined at 400 °C, 500 °C, 600 °C, and 700 °C.

The surface-to-band gap energy ratio (S/E) was calculated to determine the optimal ratio for achieving the maximum rate of RhB breakdown, as outlined in Table 3. This strategic analysis recognizes the critical interplay between surface properties and band gap energy in influencing the effectiveness of the degradation process.

Table 3. Optical properties of the synthesized materials.

Calcination Temperature	Wavelength of Light Required for Photoactivation (nm)	Band Gap (eV)	S/E Ratio
ZnO-400	371	3.153	3.982
ZnO-500	372	3.139	3.419
ZnO-600	375	3.128	2.917
ZnO-700	376	3.125	2.699

3.2. Adsorption Test

The interaction between ZnO nanoparticle samples and Rhodamine B was examined in the absence of light, and the results are illustrated in Figure 8. The adsorption capacity of ZnO nanoparticles decreases with an increase in calcination temperature. This decrease can be attributed to various factors, including heightened agglomeration (as evident in SEM data), reduced specific surface area (confirmed in Figure 2), and diminished dispersion. Moreover, the structural transformation of ZnO nanoparticles during calcination led to a reduction in surface defects [8,37], oxygen vacancies, and surface hydroxyl groups. These defects and functional groups play a fundamental role in the adsorption process, emphasizing that the control of calcination temperature enables the tailoring of ZnO materials with specific and customized adsorption capabilities.

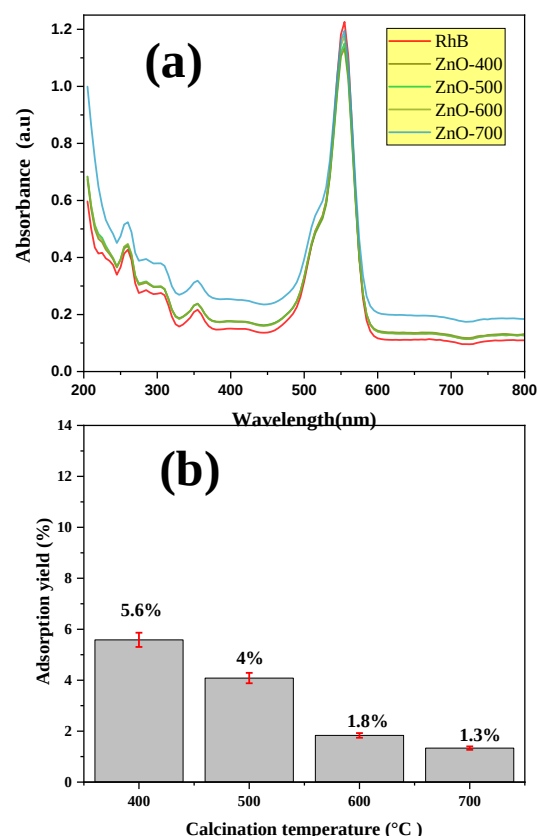


Figure 8. Adsorption capacity of ZnO calcined at different temperature for Rhodamine B at different calcination temperatures (pH = 6, S/L = 50 mg/100 mL, C_0 = 5 mg/L, time = 60 min).

3.3. Photocatalysis Tests

To evaluate the effect of calcination temperature and light source on the optical performance of ZnO, the photoactivity includes the study of the photolysis of different sources of light of Rhodamine beta. As shown in (Figure 9), the study revealed that under different

light sources (sunlight, UV, and visible light), the photoactivity of ZnO changed differently with the increase of calcination temperature from 400 °C to 700 °C from one type of light source to another. Under UV light, the photoactivity of ZnO samples showed a significant decrease with the increase of calcination temperature from (92%) at 400 °C to (27%) at 700 °C (Figure 9b). The significant decrease is related to the decrease in the area effect ratio compared to the gap effect (S/E) with the increase in calcination temperature. At 400 °C, the high S/E ratio results in a high surface area with a large availability of active sites, making ZnO very efficient under UV light due to both the ideal band gap and area. As the calcination temperature increases to 700 °C, the particle size increases as confirmed in XDR and SEM, which greatly reduces the surface area ratio and results in fewer active sites [36] for UV-driven photocatalysis, while also narrowing the band gap. The narrowing of the band gap at high temperatures is not beneficial for UV-driven photocatalysis, in contrast to visible light [66], where we observe that the low S/E ratios of ZnO-600 and ZnO-700 have efficiencies similar to those observed at higher ratios at 400 °C for visible light activity of (24%) at 700 °C to (16%) at 400 °C (Figure 9a), which is explained by the narrowing of the band gap allowing ZnO nanoparticles to partially utilize visible light for photocatalytic reactions, facilitated by localized defect states [67], where even minor absorption in the visible spectrum results in measurable photocatalytic activity [68,69]. However, the overall efficiency remains low due to the limited area. In contrast, sunlight includes all wavelengths of UV and visible light [70], allowing both S and E to influence performance simultaneously, thus mitigating the large decrease in photocatalytic efficiency observed with UV alone. The moderate decrease in the slope from 91% at 400 °C to 61% at 700 °C (Figure 9c, Table 4) indicates a compensatory effect between S and E. The slight narrowing of the band gap at higher temperatures enhances visible light absorption, resulting in stable sunlight efficiency despite reduced active sites. In addition to utilizing a wide range of wavelengths provided by natural sunlight [28], the photocatalytic charging efficiency is significantly improved, several critical factors also contribute to this phenomenon. One such factor is the presence of an aerobic environment, where oxygen acts as a redox mediator and an efficient electron acceptor [71], which resulted in the double performance in sunlight compared to other sources, where samples were exposed to UV radiation for two hours to achieve similar results like under sunlight, which was within one hour (the case of the sample calcined at 400 °C). The S-to-E ratio (S/E) is a key criterion for evaluating photocatalytic performance, integrating the effects of surface area and band gap energy.

Table 4. Kinetics parameters for the degradation of RhB dye under UV, visible, and sunlight irradiation.

Samples	k (min ^{−1})	R ²	D (%)
Under visible light			
ZnO-400	0.0045	0.909	24
ZnO-500	0.0018	0.933	20
ZnO-600	0.0017	0.965	18
ZnO-700	0.0017	0.844	16
Under sunlight			
ZnO-400	0.01492	0.984	91
ZnO-500	0.00964	0.975	78
ZnO-600	0.00781	0.980	73
ZnO-700	0.00218	0.862	61
Under UV light			
ZnO-400	0.0231	0.885	92
ZnO-500	0.0331	0.978	70
ZnO-600	0.0245	0.990	63
ZnO-700	0.0153	0.870	27

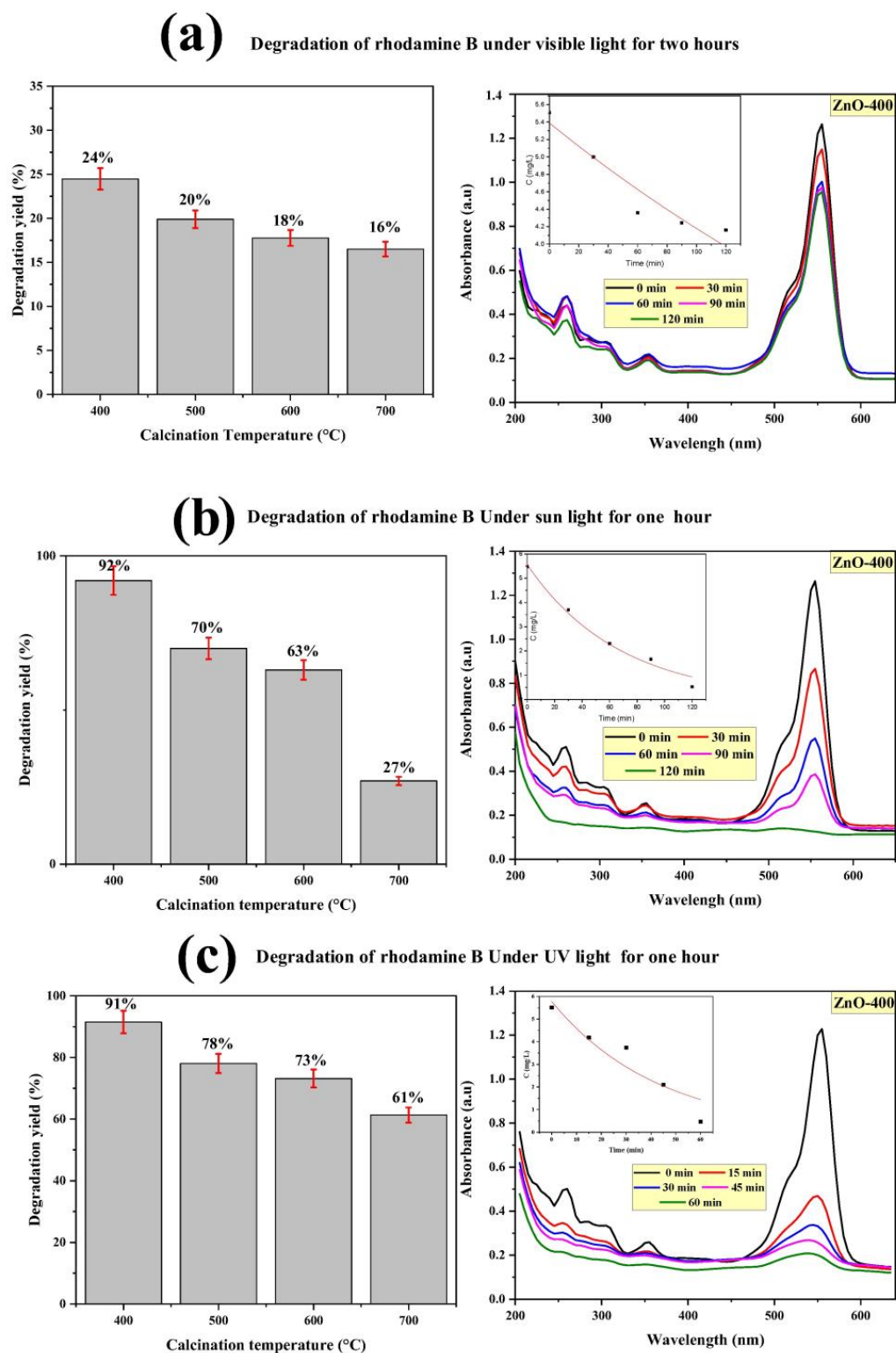


Figure 9. Degradation of RhB under visible (a), UV (b), and sunlight (c) irradiation (pH = 6, S/L = 50 mg/100 mL, C_0 = 5 mg/L, time = 60 min).

The following reactions (Equations (5)–(11)) and Figure 10 are suggested for the photodegradation of RhB by ZnO:



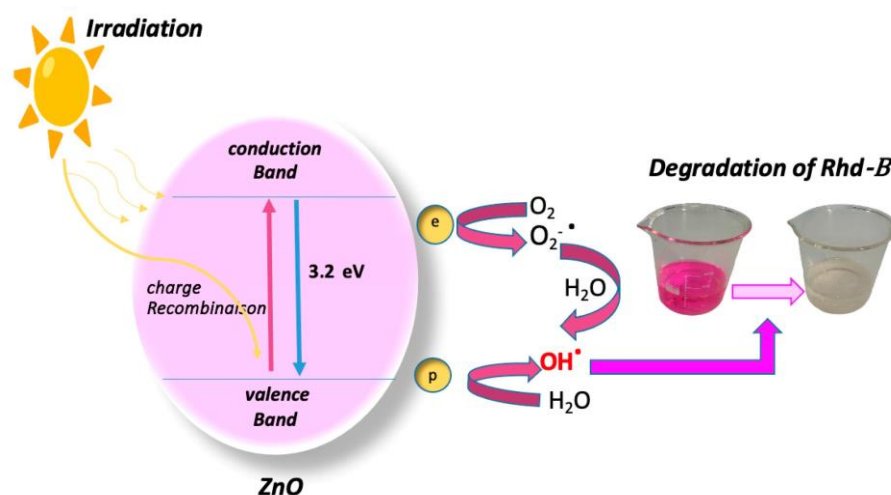
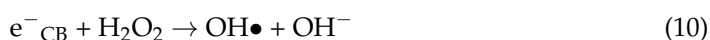
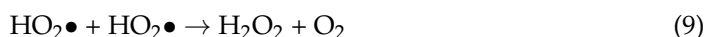
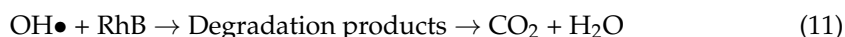


Figure 10. Degradation mechanism of RhB by ZnO.

The electrons and holes formed during the reaction in Equation (5) migrate to the surface and can react with H_2O and O_2 to selectively produce $\text{OH}\bullet$ radicals:



The $\text{OH}\bullet$ radicals further react with Rhodamine B:



3.4. Optimization

The ZnO sample calcined at 400 °C was selected for optimization experiments as it exhibited the best combination of surface area and band gap energy, making it highly effective for photocatalytic activity. Sunlight was chosen as the light source for these experiments due to its superior performance compared to other sources. Its broad wavelength spectrum, encompassing both UV and visible light, enhances photocatalytic efficiency, and its availability as a natural, sustainable energy source makes it an ideal choice.

To further optimize the performance of this sample, additional experiments were conducted by varying parameters such as catalyst mass, pH, and the concentration of H_2O_2 . These optimization steps aim to maximize the degradation efficiency of Rhodamine B under sunlight, ensuring the ZnO photocatalyst performs at its full potential.

3.4.1. Effect of ZnO Amount

The data show a correlation between ZnO amount and RhB degradation efficiency. Degradation rates increase from 56% (solid-to-liquid ratio (S/L) = 10 mg/100 mL) to 84% (S/L = 50 mg/100 mL), stabilizing at around 85% when the weight reaches 100 mg in 100 mL of Rhodamine B solution, as shown in (Figure 11a). This observed pattern suggests that increasing the mass of ZnO results in a more pronounced enhancement in photocatalytic degradation, likely attributed to the availability of more active sites for photocatalytic activity. However, the incremental rise from 50 mg to 100 mg indicates that the degradation rate has reached a limit value, light penetration limitations, particle

agglomeration, competing reactions, and diffusion limitations [72], indicating that 50 mg of ZnO is the most effective dosage for achieving the highest level of degrading efficiency. This result carries practical implications, underscoring the importance of optimizing the ZnO loading for both cost-effectiveness and efficient removal of pollutants.

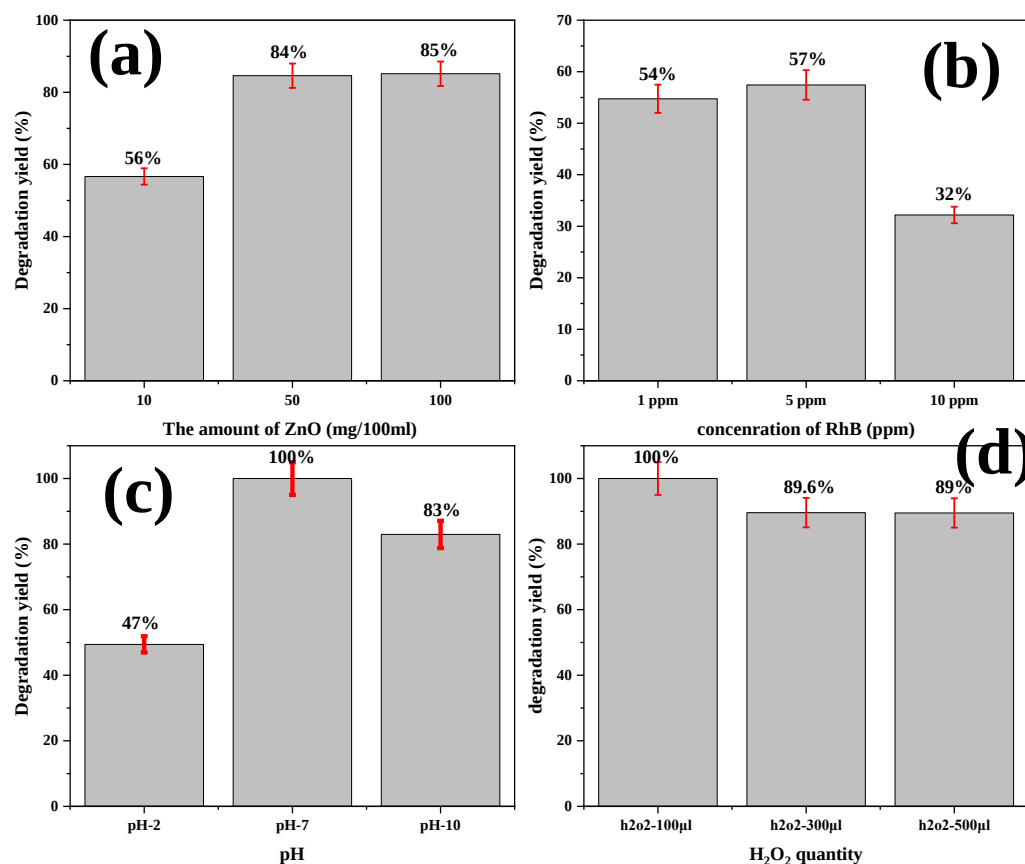


Figure 11. (a) Mass effect of ZnO degradation of RhB (pH = 6, C_0 = 5 mg/L), (b) effect of initial concentration of RhB on degradation rate (pH = 6, S/L = 50 mg/100 mL), (c) pH Effect for the degradation of RhB by ZnO under sunlight (S/L = 50 mg/100 mL, C_0 = 6 mg/L), and (d) H₂O₂ effect on the degradation of RhB (pH = 7.4, S/L = 50 mg/100 mL, C_0 = 6 mg/L).

3.4.2. Effect of the Rhodamine B Concentration

Figure 11b show the effect of varying the initial concentration of RhB on the photodegradation yield. The data reveal a relationship between the concentration of RhB and its degradation efficiency by ZnO. As the concentration increases from 1 ppm to 5 ppm and further to 10 ppm, a significant variation in the degradation yield is observed, decreasing from 54% to 32% at the maximum concentration (Figure 11b). This would suggest that higher initial concentrations of RhB caused a reduction in the efficiency of ZnO-mediated degradation. Upon closer examination of the yield at 1 ppm and 5 ppm, a slight difference becomes apparent, with yields of 54% and 57%, respectively. Although the yield is similar, the slightly elevated degradation yield at 5 ppm implies that this concentration would be somewhat more efficient than 1 ppm under the provided circumstances because, at 5 ppm, RhB molecules provide an optimal balance of reactants, allowing more species for interaction with ZnO catalysts. Efficiency in photocatalysis depends on reactant availability and active sites on the catalyst surface. Lower concentrations may lead to underutilization of the catalyst capacity; in contrast, higher concentrations of pollutant molecules at higher densities might result in a light-blocking phenomenon, reducing the entry of light into the solution and affecting the photocatalytic activity. The marginal disparity in degradation rates among these concentrations suggests that ZnO may effectively decompose RhB even

at lower concentrations, though a slight increase to 5 ppm seems to enhance the degradation process. This finding emphasizes the importance of precisely adjusting the initial concentration to achieve the possible results in studies using ZnO-mediated degradation.

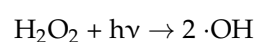
3.4.3. pH Effect

The pH in the reaction environment plays a crucial role in determining the degradation of Rhodamine B through ZnO photocatalysis, because the electrostatic interactions between ZnO nanoparticles and Rhodamine B molecules are closely linked to their charges, where the pH of the solution can play a double role in the electrostatic interactions between the solid and the dye: on the one hand, the charges on the surface of the solid change at the point of zero charge (pH_{zpc}) and, on the other hand, the dye itself takes on different forms depending on the pH relative to its pK_a [73]. We found $\text{pH}_{\text{zpc}} = 7$ for ZnO (Figure S1), in accordance with the literature [74]. Additionally, we have a positively charged species RhB^+ with, for $\text{pH} < 3.22$ (with $=\text{N}^+\text{Et}_2$) and a zwitterion $\text{RhB}^\pm$ (with $=\text{N}^+\text{Et}_2$ and COO^-) for $\text{pH} > 3.22$ [72]. At pH values higher than pH_{zpc} , the surface of ZnO tends to acquire a negative charge, enhancing its ability to attract and bond with Rhodamine B molecules, which are positively charged. This phenomenon occurs during the adsorption process, similar to what happens at a lower pH of 7 (Figure 11c). However, it is important to note that excessively elevated pH levels, reaching pH 10, reduce the photocatalytic effectiveness of the ZnO surface. Excessive pH conditions might lead to an abundance of hydroxide ions (OH^-) in the system, resulting in an environment with a large negative charge that can interact with and potentially capture the positively charged holes (h^+) [6], causing the electron–hole couples to become unstable. The literature emphasizes that pH 7 is the ideal acidity level for achieving an efficient breakdown of RhB [75,76]. Even in this experimental research, the optimal efficiency of ZnO photocatalysis for degrading RhB is achieved by the careful balance of surface charges, photocatalytic activity, and reactive oxygen species (ROS) dynamics at $\text{pH} = 7$. At this specific pH level, the interactions between ZnO and Rhodamine B are optimized, resulting in a robust adsorption process and concurrently facilitating the production of $\text{OH}^\bullet$ radicals.

4. Effect of H_2O_2 Dose

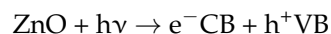
The interaction of H_2O_2 with ZnO leads to complex formation, increasing the surface area of ZnO nanoparticles and offering additional active sites for photocatalytic reactions. The enhanced surface area, resulting from the complex structure [77], facilitates a greater likelihood of interactions, leading to effective photocatalysis. The process requires an optimal concentration of H_2O_2 (Figure 11), as evidenced by studies indicating that 100 μL of H_2O_2 markedly improves photocatalytic activity in the degradation of Rhodamine B (RhB). Higher concentrations, specifically 300 μL and 500 μL , do not lead to a proportional increase in reactive oxygen species (ROS) generation, suggesting the existence of a saturation point where competition for active sites restricts further advantages [6]. This equilibrium indicates an optimal balance that enhances the efficiency of ROS generation while preventing excess H_2O_2 from hindering the reaction.

The generation of hydroxyl radicals ($\cdot\text{OH}$) from H_2O_2 is fundamental to this enhanced degradation process and can be expressed by the reaction:

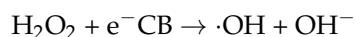


In this context, $h\nu$ denotes photons possessing adequate energy to activate the ZnO photocatalyst, thereby commencing the reaction. When exposed to UV or sunlight, ZnO

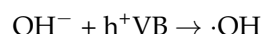
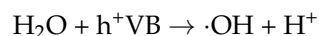
absorbs photons with energy at or above its band gap, resulting in the excitation of electrons (e^-) from the valence band to the conduction band, thereby generating holes (h^+):



The conduction band electrons interact with H_2O_2 to produce hydroxyl radicals:



Simultaneously, the valence band holes oxidize water (H_2O) or hydroxide ions (OH^-) to generate additional hydroxyl radicals [78]:



These hydroxyl radicals are highly reactive and attack the chromophore structure of RhB, initiating its breakdown into intermediate products, which are further degraded into CO_2 and H_2O :



The combined mechanism elucidates the high efficiency noted in the $\text{H}_2\text{O}_2 + \text{ZnO}$ system, resulting in complete degradation within 25 min of sunlight exposure.

5. Scavenger Effect

This part of our work examines the impact of scavenger species, namely methanol and benzoquinone, on the effectiveness of ZnO photocatalysis in breaking down RhB (Figure 9). This study seeks to clarify the precise processes involved in pollutant degradation and provide insights for enhancing photocatalytic systems for environmental remediation applications by understanding the influence of these scavengers on the photocatalytic process.

The exceptional efficacy of ZnO photocatalysis in breaking down RhB under the stated circumstances is highlighted by the total destruction of the compound in the absence of scavengers. Nevertheless, the recorded decrease of 30% in degradation when methanol is present indicates that methanol behaves as an electron scavenger, hence exerting a detrimental influence on the photocatalytic process by constraining the accessibility of electrons. Furthermore, it is well recognized that methanol effectively scavenges $\cdot\text{OH}$ radicals, whereas benzoquinone effectively scavenges $\text{O}_2^{\bullet-}$ [79]. The histogram presented in Figure 12 indicates that the $\cdot\text{OH}$ radicals demonstrate the highest level of significance. The incorporation of benzoquinone results in a slightly increased degradation rate of 55%, indicating that, although it acts as a scavenger, its effect is relatively less significant than that of methanol. Benzoquinone selectively eliminates or accumulates holes, thereby promoting segregation between electrons and holes, which ultimately leads to the generation of reactive oxygen species (ROS) [80].

In order to evaluate the degradation efficiency of the ZnO synthesized in this study, we compared it to that of ZnO reported in the literature. Table 5 provides a comparison of the key findings from our study with those reported in the literature.

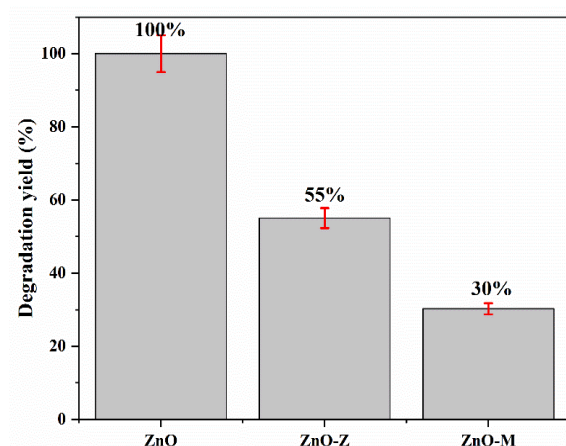


Figure 12. Scavenger effect on photodegradation of RhB by ZnO under sunlight, pH = 6, S/L = 50 mg/100 mL, $C_0 = 3$ mg/L and [Scavenger] = 1×10^{-5} M.

Table 5. Comparison of different studies on the degradation of Rhodamine B by ZnO prepared with different methods.

Preparation Method	Focus	Light Source	Condition of Photocatalysis	D (%)	Reference
green synthesis method using <i>Argyrea nervosa</i> leaf calcined at 500 °C, 4 h	The focus is on the biosynthesis of ZnO nanoparticles using <i>Argyrea nervosa</i> leaf extract and their photocatalytic efficiency in removing Rhodamine B dye	visible (100 W)	C_0 : 5 ppm pH = 11 S/L = 1.4 g/L	48% in 180 min	[78]
sol-gel synthesis technique calcined from 190 to 550 °C	The study focuses on the effect of calcination temperature on the UV light-driven photocatalytic degradation of Rhodamine B by ZnO nanoparticles	UV	C_0 : 25 ppm pH: 7 S/L = 2 g/L	95% in 120 min	[79]
Sol-gel calcined at different temperatures 400 °C	The focus of the study is on the effect of calcination temperature in photocatalytic properties of ZnO nanoparticles under UV light	UV (330 W)	C_0 : 10 ppm S/L: 2 g/L	95% in 160 min	[80]

Table 5. Cont.

Preparation Method	Focus	Light Source	Condition of Photocatalysis	D (%)	Reference
Green synthesis using the leaf extract of <i>Cyanometraramiflora</i>	ZnO nanoparticles synthesized using the leaf extract of <i>Cyanometra ramiflora</i> demonstrated effective photocatalytic degradation of Rhodamine B dye under sunlight, showcasing their potential for eco-friendly and sustainable dye remediation	sun light	C ₀ : 10 ppm S/L = 20 mg in 100 mL of solution	98% in 200 min	[81]
hydrothermal method	This study highlights a simple hydrothermal method to synthesize ZnO microcrystals with high photocatalytic efficiency and easy recovery, optimized for sustainable industrial applications under uv	UV (15 w)	C ₀ :10 ppm S/L: 1 g/L	97% in 40 min	[82]
Precipitation Calcined 400 °C to 700 °C	Our study demonstrates the optimized photocatalytic performance of ZnO under UV, visible, and solar light by introducing the S/E metric and leveraging controlled precipitation and calcination; optimization of the samples calcined at 400 °C under sun light	sun light uv light visible light	C ₀ : 5 ppm sun light S/L: 0.5 g/L	92% in 60 min	This study

6. Conclusions

Renewable natural sunlight is a more effective and ecologically sustainable energy source for photocharging applications compared to standard artificial light sources such as visible and UV light. Our results are based on a thorough investigation highlighting the higher photocatalytic performance of zinc oxide (ZnO) nanoparticles under natural sunlight conditions; this stresses the benefits of using solar energy and showcases the impressive capability of ZnO to use various sunshine wavelengths. The work improves the capability for removing pollutants and creates a more realistic testing setting for measuring photocatalytic effectiveness by using natural sunshine conditions. The study provides interesting information on the ideal circumstances for ZnO nanoparticle efficiency. Zinc oxide samples heated to 400 °C provide the best results. This temperature enhances crystal formation, improves surface area, and promotes the effective creation and usage of electron–hole pairs under light, while it is important to recognize that higher temperatures result in decreasing reactivity, emphasizing the intricate relationship among calcination temperature, surface properties, and photocatalytic effectiveness in ZnO nanoparticles. Where the study found peak decomposition efficiency correlates with the highest surface area to gap energy ratio, emphasizing surface optimization’s importance

For applications that rely on UV or sunlight, maintaining a high S/E ratio is critical. Low calcination temperatures can preserve surface area and active sites, thereby maximizing photocatalytic efficiency. Conversely, visible light applications are enhanced by

methods that lower the band gap energy, including doping or the formation of hybrid structures. These methods improve performance despite a reduced S/E effect, facilitating the creation of specialized ZnO materials designed for photonic applications.

Comprehending and enhancing these elements are crucial for creating very effective photocatalytic devices for practical use in environmental cleanup and sustainable energy generation. In addition, in this work, the effect of hydrogen peroxide (H₂O₂) on the degradation of RhB was studied. It was shown that H₂O₂ was a very good oxidant for improving the degradation of RhB under sunlight. ZnO has great promise for tackling environmental issues and promoting green technology by using the plentiful and clean energy from sunshine. Continuing study in this area might result in the creation of more effective and expandable photocatalytic devices, which would help create a more sustainable and environmentally friendly future.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/w17010032/s1>. Table S1: Effect of H₂O₂ addition on the kinetic parameters for the degradation of RhB dye under sunlight. Table S2: Effect of solution pH on the kinetic parameters for the degradation of RhB dye under sun light. Figure S1: PZC of ZnO Nanoparticles Determined by pH Titration.

Author Contributions: Conceptualization, A.S., R.B., L.B. and K.F.; methodology, A.S., R.B., K.F., L.M., A.B. and A.D.; software, R.B. and A.B.; validation, L.M., J.-C.B. and S.S.; formal analysis, S.S., L.M. and R.B.; investigation, A.A.A. and A.S.; resources, A.A.A., L.B. and K.F.; data curation A.S. and R.B.; writing—original draft preparation, A.S., R.B., K.F. and L.B.; writing—review and editing, L.M., J.-C.B., A.A.A. and S.S.; visualization, L.M., J.-C.B., S.S. and A.B.; supervision, A.B., L.M. and J.-C.B.; project administration, K.F., A.B. and K.F. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Researchers Supporting Project number (RSPD2024R527), King Saud University, Riyadh, Saudi Arabia.

Data Availability Statement: Data is contained within the article.

Acknowledgments: The authors acknowledge the financial support through the Researchers Supporting Project, number (RSPD2024R527), King Saud University, Riyadh, Saudi Arabia.

Conflicts of Interest: The authors declare no conflict of interest.

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