jurnal βeta kimia
e-ISSN: 2807-7938 (online) dan p-ISSN: 2807-7962 (print)
Volume 5, Number 1, May 2025
http://ejurnal.undana.ac.id/index.php/jbk

Low-Temperature Synthesis of ZnO Nanoparticles Using a Water-Methanol Solvent for Rhodamine B Photodegradation

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ARTICLE INFO

Article history:
Received:
9 April 2025
Revised:
25 April 2025
Accepted:
27 April 2025

Keywords:
Low-temperature, photodegradation, photocatalyst, water-methanol solvent, ZnO nanoparticles.

ABSTRACT

Water pollution caused by organic dyes represents a significant environmental issue demanding effective treatment methods. This study aimed to synthesize zinc oxide (ZnO) nanoparticles at a low temperature using a water-methanol mixed solvent and evaluate their photocatalytic potential for the degradation of Rhodamine B (RhB). ZnO nanoparticles were successfully synthesized at a low temperature of 60°C utilizing a mixture of water and methanol as the solvent. Characterization results from X-ray diffraction (XRD) and Field Emission Scanning Electron Microscopy (FESEM) confirmed that the synthesized material consists of wurtzite structure ZnO nanoparticles. Analysis using UV-Vis Diffuse Reflectance Spectroscopy (DRS) showed that the material possesses a band gap energy of 3.16 eV. The as-synthesized ZnO nanoparticles exhibited effective photocatalytic activity for the degradation of the model pollutant RhB under UV light irradiation. A high photodegradation efficiency of 95.58% was achieved within 45 minutes, with a first-order reaction rate constant (k) of 0.0571 min^{-1}. This low-temperature synthesis method based on the water-methanol solvent mixture proved to be a promising and efficient route for producing active ZnO photocatalysts potentially applicable in treating organic dye-contaminated wastewater.

How to cite: Christanti, C.C., Benu, D.P. (2025). Low-Temperature Synthesis of ZnO Nanoparticles Using a Water-Methanol Solvent Mixture for Rhodamine B Photodegradation, 5(1), 62-69. https://doi.org/10.35508/jbk.v5i1.21305

INTRODUCTION

Water pollution by organic compounds, particularly dyes from the textile, chemical, and pharmaceutical industries, is a serious global environmental issue [1]. Wastewater containing organic dyes such as Rhodamine B exhibits toxic characteristics, persistence, difficulty in natural degradation, and can inhibit light penetration into water bodies, disrupting aquatic ecosystems [2-3]. Conventional wastewater treatment methods, such as adsorption, coagulation-flocculation, and filtration, only transfer pollutants from one phase to another or fail to degrade complex compounds completely [4].

In recent decades, Advanced Oxidation Processes (AOPs) have attracted attention as a promising alternative for wastewater treatment. AOPs operate by generating highly reactive free radical species (e.g., hydroxyl radicals •OH) that are capable of degrading organic pollutants into harmless or less toxic end products, even to total mineralization [5-6]. Among various AOP techniques, semiconductor photocatalysis is considered one of the most environmentally friendly and sustainable methods, as it utilizes light energy (UV or visible light) as a source of energy and a recyclable catalyst [7-8]. Various semiconductor materials have been explored as

photocatalysts, including TiO_2, ZnO, CdS, and others. Among these materials, Zinc Oxide (ZnO) stands out due to several advantages, including a relatively wide band gap (~3.37 eV), high exciton binding energy (~60 meV), non-toxicity, abundance in nature, and relatively low production cost [9-10]. ZnO also exhibits high photocatalytic activity for the degradation of various organic pollutants [7, 11].

Semiconductor materials are often synthesized at the nanoscale (nanoparticles) to enhance photocatalytic performance. ZnO nanoparticles have a much larger specific surface area compared to micron-sized materials, providing more active sites for photocatalytic reactions. Additionally, the nanoscale size can affect the optical and electronic properties of the material, potentially improving the efficiency of electron-hole pair separation generated by light irradiation [10, 12]. The properties and performance of ZnO nanoparticles highly depend on the synthesis method used, such as coprecipitation, hydrothermal, sol-gel, solvothermal, and others [9-10]. Many synthesis methods require high temperatures or the use of specific and sometimes hazardous organic solvents. Therefore, there is a need to develop more efficient, environmentally friendly synthesis methods for ZnO nanoparticles under operational conditions that are more economical (such as low temperatures).

The use of solvent mixtures, such as water and methanol, in nanoparticle synthesis can offer advantages. This solvent mixture not only affects the solubility of the precursors but can also modify the reaction environment, control the crystallization kinetics, and influence the morphology and size of the formed particles [13-14]. Synthesis at low temperatures can reduce energy consumption and simplify the process, making it more economical and scalable [15-16]. An in-depth exploration of the specific effects of water-methanol solvent mixtures at low temperatures on the characteristics of the resulting ZnO nanoparticles and their correlation with photocatalytic performance is still needed. Given the challenges in the treatment of organic dye waste, the potential of ZnO as a photocatalyst, and the potential benefits of low-temperature synthesis methods using a water-methanol solvent mixture, this study focuses on synthesizing ZnO nanoparticles using this method. Rhodamine B was chosen as a model pollutant because it is a common, persistent cationic dye frequently used as a benchmark standard for evaluating photocatalytic performance [17-8].

This article reports the synthesis of ZnO nanoparticles using a water-methanol solvent mixture at low temperature, characterizing the structure, morphology, and optical properties of the resulting material and evaluating its effectiveness as a photocatalyst in degrading Rhodamine B under UV light irradiation. The novelty of this research lies in the composition of the water-methanol solvent mixture and the low-temperature synthesis conditions (60°C). It is expected that the developed synthesis method will produce an effective ZnO photocatalyst and contribute to the development of more efficient and sustainable wastewater treatment technologies.

MATERIALS AND METHODS

Materials

The materials used in this study are zinc acetate dihydrate (99%, Merck), methanol (99.8%, Merck), deionized water, and technical ethanol. The main material for the synthesis of ZnO was used directly without any purification process.

Synthesis of ZnO Nanoparticles

ZnO nanoparticles were synthesized at a low temperature in a water-methanol solvent mixture. A total of 1.0975 g of $Zn(CH_3COO)_2{\cdot}2H_2O$ was added to 54 mL of methanol and stirred until a homogeneous solution was formed. To this solution, 6 mL of deionized water was added and stirred for 30 min. The resulting mixture was reacted at 60°C for 16 h using an oil bath. After the reaction time, the reaction vessel was allowed to cool to room temperature. The resulting solid

was separated using filter paper, rinsed consecutively with deionized water and ethanol, and then dried in an oven at 60°C for 12 h.

Characterization of ZnO Nanoparticles

The particle structure of the synthesized ZnO was characterized using X-ray diffraction (XRD). The morphology of the particles was characterized using a Field Emission Scanning Electron Microscope (FESEM). The optical properties of the particles were characterized using UV-Vis Diffuse Reflectance Spectroscopy (DRS). From this data, the band gap energy was determined using a Tauc plot based on the Kubelka-Munk function.

Photocatalytic Activity Test of ZnO Nanoparticles

The photocatalytic activity of the synthesized ZnO was evaluated in the photodegradation reaction of Rhodamine B, which was used as a model pollutant dye solution. A total of 50 mg of ZnO nanoparticles was dispersed into 50 mL of Rhodamine B solution (5 ppm). The mixture was then stirred continuously in the dark using a magnetic stirrer for 15 minutes. Subsequently, the mixture was irradiated with a UV-LED lamp (λ = 365 nm, P = 3 W, $\phi = 3\times10^4$ W m^{-2}) for 45 min. A 5 mL sample of the Rhodamine B solution was taken every 15 minutes, separated from the ZnO particles, and then analyzed using UV-Vis spectroscopy. The kinetics of the photodegradation reaction were calculated using the following first-order reaction rate Eq. 1:

$$\ln(\frac{A_t}{A_0}) = -kt \quad (1)$$

where A_t is the absorbance of the Rhodamine B solution at time t, A_0 is the absorbance of the Rhodamine B solution after stirring in the dark, t is the reaction time, and k is the reaction rate constant. By plotting the value of ln (A_t/A_0) on the y-axis and t on the x-axis, the slope obtained represents the rate constant (k). Additionally, the photocatalytic efficiency was calculated using the following Eq. 2:

$$\eta(t) = \frac{(A_0 - A_t)}{A_0} \times 100\% \quad (2)$$

where $\eta(t)$ is the photocatalytic efficiency at time t.

RESULTS AND DISCUSSION

The success of the ZnO synthesis was confirmed through analysis using X-ray diffraction (XRD). Figure 1 presents the diffraction pattern of the synthesized particles, which corresponds to the Bragg positions of ZnO (CIF 2300450), along with an image of the unit cell of the wurtzite ZnO crystal structure [19]. The diffraction pattern in Figure 1a exhibits peaks that match with the reference Bragg positions for ZnO. The result is characterized by three distinct diffraction peaks: the (100) peak at 2θ = 31.8°, the (002) peak at 2θ = 34.5°, and the (101) peak at 2θ = 36.3°, along with other peaks that match the reference Bragg positions. The result confirms that the synthesized particles are ZnO with a wurtzite structure. Furthermore, no additional peaks from impurities were observed, indicating that the synthesized product is pure ZnO with a wurtzite crystal structure. Figure 1b illustrates the atomic arrangement in the wurtzite ZnO structure. In this structure, each zinc (Zn) atom is coordinated with four oxygen (O) atoms, and each oxygen atom is similarly coordinated with four zinc atoms. These bonds form a fundamental tetrahedral unit, and the Zn-O and O-Zn tetrahedra are interconnected throughout the crystal by sharing their corner points. This regular corner-sharing arrangement creates a characteristic three-dimensional network with a hexagonal pattern [20], [21].

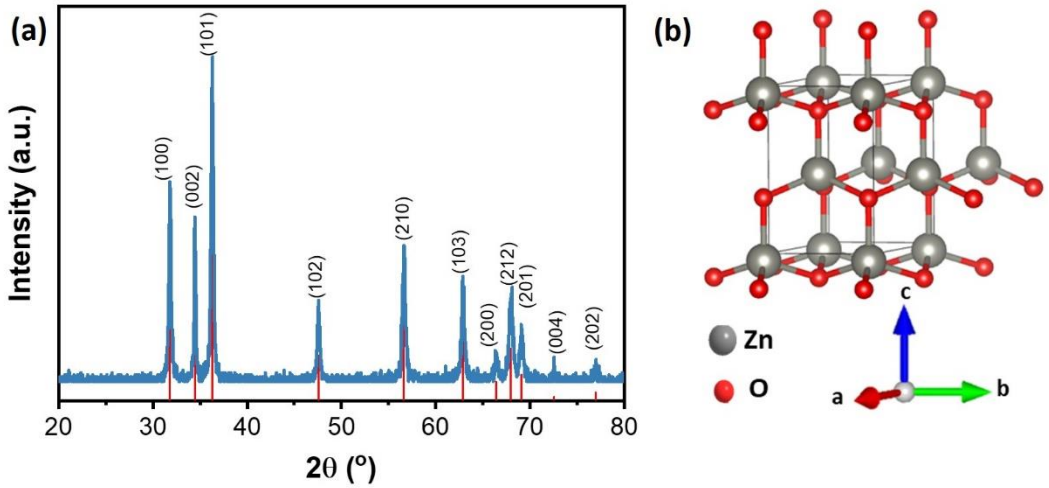

Figure 1. (a) XRD pattern of the synthesized particles. The colored drop lines represent the Bragg positions of wurtzite-structured ZnO (space group P63mc) based on CIF data 2300450 [19]. (b) Crystal unit cell of wurtzite ZnO extracted from CIF 2300450 [19].

The particles were observed using Field Emission Scanning Electron Microscopy (FESEM) to confirm that the synthesized ZnO consists of nanoparticles. Figure 2 shows the SEM images of the synthesized particles. At low magnification, small particles with relatively uniform sizes can be observed (Figure 2a). The high-magnification SEM image in Figure 2b reveals the shape and size of the synthesized ZnO nanoparticles. These particles have an approximate size of ±100 nm and exhibit a slightly elongated hexagonal shape. This hexagonal morphology is characteristic of the wurtzite ZnO crystal habit, which typically forms rapidly along the c-axis, resulting in hexagonal rods. The low aspect ratio between the length and diameter of the produced ZnO nanoparticles suggests that the synthesis method successfully inhibited the growth of ZnO crystals along the c-axis.

In the application of ZnO in the field of photocatalysis, one important factor to consider is the interaction of the material with UV-Vis light. Therefore, the optical properties of the synthesized ZnO nanoparticles were characterized using UV-Vis Diffuse Reflectance Spectroscopy (DRS). Figure 3 shows the absorption spectrum of the ZnO nanoparticles along with the Tauc plot to determine the band gap energy (Eg). The absorption spectrum in Figure 3a shows a sharp absorption in the wavelength range of 400 nm. This absorption corresponds to electron transitions from the valence band to the conduction band in a semiconductor material with a wide band gap [22], [23]. The band gap energy value was determined through the Tauc plot based on the Kubelka-Munk equation for direct band gap [24], as shown in Figure 3b. From

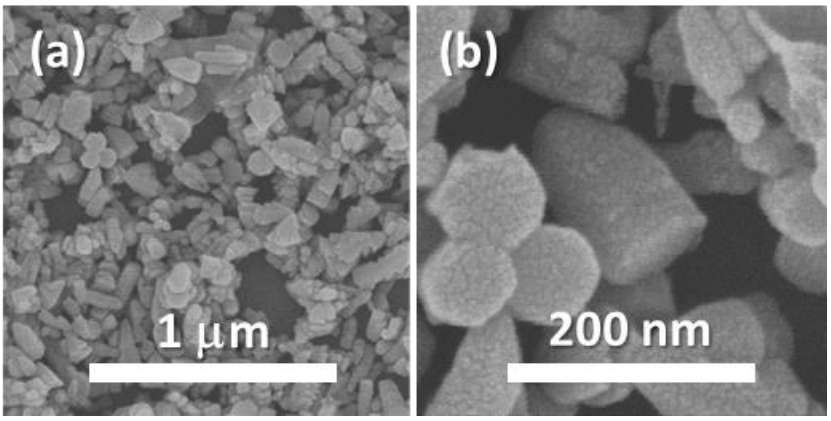

Figure 1. SEM images of the synthesized particles; (a) low magnification, (b) high magnification.

the Tauc plot, the band gap energy of the synthesized ZnO nanoparticles was found to be 3.16 eV. This band gap energy corresponds to a photon wavelength of 392 nm. Additionally, the minimal curvature in the absorption edge region indicates that the ZnO nanoparticles produced have a low crystal defect density [11]. Therefore, it can be concluded that the synthesized ZnO nanoparticles can perform well as a photocatalyst within the UV light range (λ<392 nm).

The photocatalytic activity of the synthesized ZnO nanoparticles was evaluated in the photodegradation of Rhodamine B dye. Figure 4 presents the results of Rhodamine B dye photodegradation using the synthesized ZnO nanoparticle photocatalyst. The photograph of the Rhodamine B solution in Figure 4a illustrates that the color of the solution gradually fades as the degradation time increases. This observation is further confirmed by the absorption spectrum of the Rhodamine B solution in Figure 4b. In general, the absorbance value of the Rhodamine B solution decreases as the irradiation time progresses.

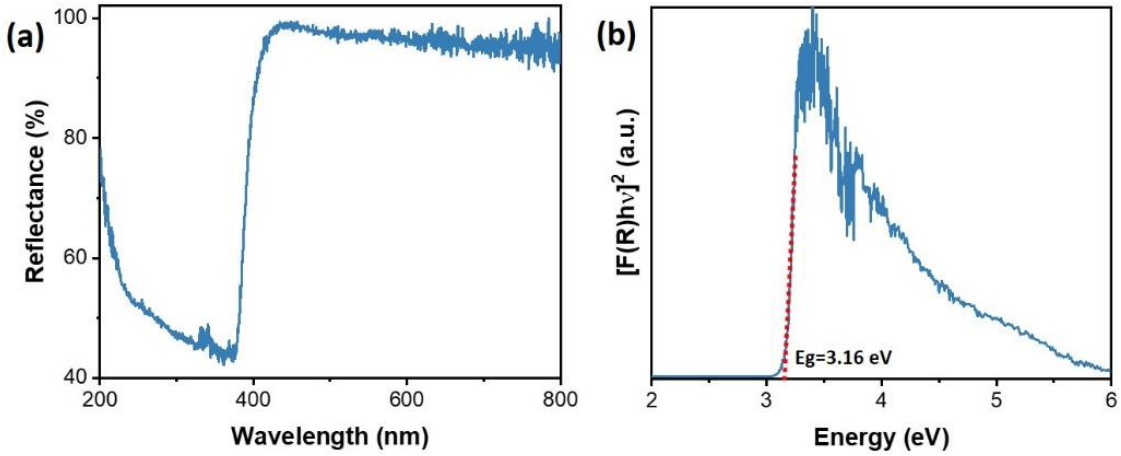

Figure 3. (a) Absorption spectrum of the synthesized ZnO nanoparticles, (b) Tauc plot to determine the band gap energy.

From the absorption spectrum data, the adsorption and photodegradation efficiencies of Rhodamine B were calculated, as shown in Figure 4c. In the dark condition, the process observed is the adsorption of Rhodamine B onto the surface of the ZnO nanoparticles. This adsorption process was able to reduce the Rhodamine B concentration by 37.29%. Under UV light irradiation, the predominant process is photodegradation. As the irradiation time increases, the amount of Rhodamine B degraded also increases. After 45 minutes of irradiation, a photodegradation efficiency of 95.58% was achieved.

In addition to calculating the photodegradation efficiency, the obtained data were further analyzed to determine the kinetic parameters of the photodegradation reaction. Figure 4d shows the first-order reaction kinetics plot for the photodegradation of Rhodamine B catalyzed by ZnO nanoparticles. The plot yields a reaction rate constant (k) of 0.0571 min^{-1}. This k value is higher than those reported in several previous studies on the photodegradation of Rhodamine B. The photocatalysts used in those studies were: (i) ZnO nanoparticles (k = 0.0538 min^{-1}) [25], (ii) rice-like ZnO (k = 0.0432 min^{-1}) [26], (iii) disk-like ZnO (k = 0.0245 min^{-1}) [26], and (iv) flower-like ZnO (k = 0.0072 min^{-1}) [27]. Therefore, the findings of this study hold potential for further development toward practical applications as a photocatalyst for the degradation of dye waste in real-world environmental conditions.

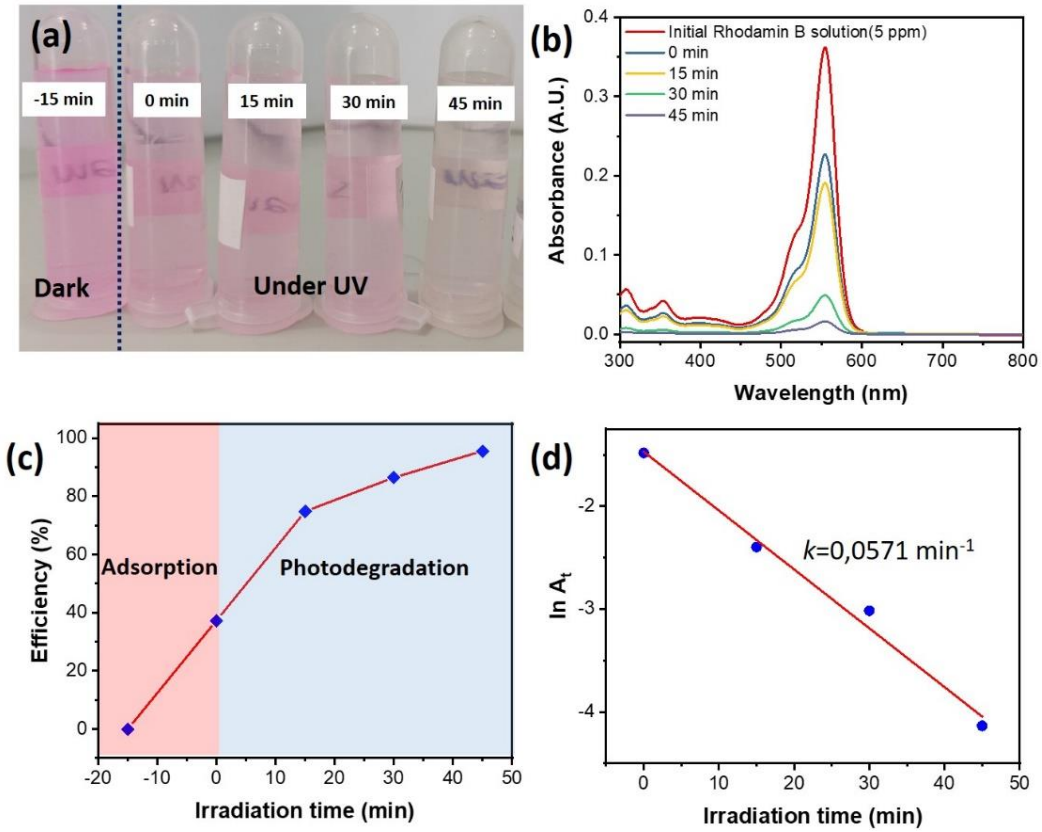

Figure 4. Photodegradation results of Rhodamine B using the synthesized ZnO nanoparticle photocatalyst; (a) photograph of the solution at each degradation time interval, (b) absorption spectrum, (c) adsorption and photodegradation efficiency, (d) first-order reaction kinetics plot.

CONCLUSION

ZnO nanoparticles have been successfully synthesized at a low temperature using a mixture of water-methanol solvent. Characterization results indicate that the synthesized material is ZnO nanoparticles with a characteristic wurtzite crystal structure and a band gap energy of 3.16 eV. The ZnO nanoparticles produced by this synthesis method exhibit effective photocatalytic activity in degrading the model pollutant Rhodamine B under UV light irradiation, with a photodegradation efficiency of 95.58% at 45 minutes and a rate constant (k) of 0.0571 min^{-1}. This low-temperature synthesis method using a water-methanol solvent mixture has proven to be a promising and efficient approach for producing active ZnO photocatalysts, with potential applications in wastewater treatment contaminated with organic dyes.

ACKNOWLEDGEMENTS

The authors would like to thank the LPPM of the University of Timor for the financial support provided for this research through the 2024 Laboratory Basic Research Scheme.

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