



## Facile Synthesis of ZnO Nanoparticles for the Photodegradation of Rhodamine-B

Tety Sudiarti<sup>a,\*</sup>, Neng Hani Handayani<sup>a</sup>, Yusuf Rohmatulloh<sup>a</sup>, Silmi Rahma Amelia<sup>a</sup>, Ravli Maulana Yusuf<sup>a</sup>, Atthar Luqman Ivansyah<sup>b,c</sup>

<sup>a</sup> Chemistry Department, Faculty of Science and Technology, UIN Sunan Gunung Djati, Bandung, Indonesia

<sup>b</sup> Master Program in Computational Science, Faculty of Mathematics and Natural Sciences, Institut Teknologi Bandung, Bandung, Indonesia

<sup>c</sup> Analytical Chemistry Division, Department of Chemistry, Faculty of Mathematics and Natural Sciences, Institut Teknologi Bandung, Bandung, Indonesia

\*Corresponding author email: [tety.sudiarti@uinsgd.ac.id](mailto:tety.sudiarti@uinsgd.ac.id)

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### Abstract

River pollution is a problem that is still very poorly handled. Industrial growth is the most significant contributor to produce this wastewater. The industry produces liquid waste such as dyes that do not meet handling standards because of the high cost. Photocatalyst is way better than other methods such as adsorption, coagulation, fluctuation, and others. However, there are still many shortcomings of the existing methods, such as high cost, high temperature, and dangerous by-products. This research seeks to provide a solution by synthesizing zinc oxide (ZnO) nanoparticles as a photocatalyst to reduce rhodamine B dye under visible light irradiation. ZnO nanoparticles were successfully synthesized through a simple sol-gel method in the form of a white powder by heating at a low temperature, 60°C. The XRD results show that the results have a diffraction peak that follows the standard ZnO with a hexagonal wurtzite crystal structure. According to the Scherrer equation, the crystal has a size of 22.61 nm. SEM analysis showed that the particle morphology and particle size were homogeneous with a spherical shape, ranging from 22-24 nm. Optimal ZnO photocatalytic activity at 90 minutes with an efficiency of 98.83%.

### 1. Introduction

The increasingly advanced era, making industry increasingly developing all over the world. The existence of industry in Indonesia brings one positive side and brings a negative side. One of the community's negative impacts that are felt most directly is the deterioration of environmental conditions caused by pollution in various aspects such as soil, water, and air pollution. Liquid waste in Indonesia is severe and has polluted the water. Rivers that are beginning to blacken with a strong odor in Indonesia indicate that the waters are polluted. This liquid waste comes from dyes from the textile industry, which uses organic compounds difficult to decompose and toxic. The presence of toxic organic substances and harmful dyes in wastewater creates a real threat to the environment. Therefore, any liquid waste needs to be

adequately treated [1, 2]. Rhodamine B, one of the textile industries wastes that pollute Indonesian waters, needs special handling because this compound is difficult to decompose and, when consumed by humans, can endanger human health. If it continues to be consumed in large doses and quite frequently, it can cause cancer. Studies about rhodamine B are still rare compared to other dyes, such as methylene blue. Therefore, this research was conducted to provide solutions to solve environmental problems caused by rhodamine B.

Various wastewater treatment and handling techniques have been developed to remove toxic contaminants from wastewater, such as adsorption [3], coagulation/flocculation [4], ozonation [5], biological treatment methods [6], photocatalysis [7], and others. One of the economical and effective ways to degrade

rhodamine B is photocatalysts [8]. One of the economical and effective ways to degrade rhodamine B is the use of a photocatalyst. Compared to other methods, photocatalyst has the advantage of not producing other wastes as happened when handling using the adsorption [3] and coagulation/flocculation method [4], which turned liquid waste into solid waste that needed further handling. Photocatalysts convert complex substances that are dangerous and difficult to decompose into compounds that are more environmentally friendly. Besides, photocatalysis is a low-cost and sustainable process for treating pollutants in water, including organic substances. In the photocatalytic degradation process, the toxic organic molecules are completely degraded through the oxidation process.

The photocatalytic process requires semiconductor compounds to produce radical compounds that will degrade the dye compound. Several compounds, especially metal oxides, have good photocatalytic activity and are being developed. Materials functional as photocatalysts include ZnO [9, 10], TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub> [11], and many researchers who make nanocomposites from several compounds to obtain better results from the primary compound. Some of them are also doped with other metals. Metal oxide compounds are classified as semiconductor compounds, for example, ZnO. ZnO can absorb ultraviolet light, which allows it to be used for the photodegradation of organic pollutants from wastewater. However, the bandgap of the ZnO bulk was not good enough as a photocatalyst under visible/solar irradiation. Consequently, it is essential to modify the bandgap through the surface layer by doping with other dye-sensitive elements or changing the size of the bulk-sized particles to form the nanoparticles [12].

The previous study synthesized ZnO nanoparticles using the sol-gel method with various calcination temperatures (400, 500, and 600°C) to photocatalyze the degradation of rhodamine B under exposure to visible light. The optimum condition was obtained at 0.2 g ZnO in 10 ppm rhodamine B, which resulted in efficient degradation of 95.41%. Efforts to maximize the photocatalytic activity of ZnO were carried out by doping ZnO with Ba and tested its activity in degrading rhodamine B under exposure to visible light. The optimum condition was obtained at 350 ppm Ba/ZnO in rhodamine B 4.11 ppm, resulting in a degradation efficiency of about 98.40% [13]. Because of its good photocatalytic activity, ZnO has attracted the attention of many researchers, so that it is widely used by several researchers around the world [14, 15].

Many methods can be used to synthesize ZnO, one of which is the sol-gel method. Many previous studies have used this method because it is easy and does not use toxic materials, nor does it produce side products that are harmful to the environment [16, 17]. Nevertheless, most of them operate at high temperatures, so they are still less efficient. The previous studies using high temperatures of 400°C [18], 500°C [19], 700°C [17, 20], and even 800°C [16]. This has an impact on high operational costs so that it cannot be said low cost. Some of them use surface modifiers and other additives to reduce particle size.

However, in this study, only a few materials were used and operated at a low temperature, but it produced a product with good activity as a photocatalyst. With this, this research can compete and can be widely applied.

The change in particle size also has a beneficial effect because the smaller the particle size will also cause an increase in the surface area, which is directly proportional to the number of reactions that occur so that the reaction results will be better. This also applies to ZnO nanoparticles with a bandgap of 3.37 eV [21] which means that they are more significant than ZnO bulk which has a bandgap of 2.40 eV [12]. In other words, the smaller the particle size, the bigger the bandgap will be. In the synthesis process, several methods can be taken to obtain these nanoparticles, including solvothermal [22], hydrothermal [23], dispersion [24], and sol-gel [25], as was done in this study. The sol-gel method was decided as the method used in this study. Compared to other methods that use high-temperature heating, this sol-gel method operates at low temperatures, resulting in homogeneous compounds, high purity, and relatively low cost.

This research aims to overcome environmental problems and focus on wastewater treatment so that it is not dangerous when it enters the environment with the proper technique that does not only change the form of waste but also provides solutions that can directly solve the problem. This study synthesized ZnO nanoparticles using a simple sol-gel method with low temperature and low cost to obtain nano-sized particles. The synthesis time is not long and does not produce harmful by-products, so it is environmentally friendly. Finally, after testing the photocatalytic activity of rhodamine B under visible light, the ZnO photocatalyst produced in this study has good optical properties as a photocatalyst indicated by its high-efficiency value.

## 2. Methodology

### 2.1. Material

The materials to be used in this research are Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O (Merck®), KOH (Merck®), methanol (pro-analysis), double distilled water, and rhodamine B (pro-analysis). XRD (PANalytical - X'Pert High Score) and SEM (JEOL JCM 6000) were used to test the synthesized products' phase, crystallinity, and surface morphology. Meanwhile, to investigate the photocatalytic activity of the synthesized substance, a sonicator (Digital Ultrasonic Cleaner 35-Watt 100-110 V, 220-240 V 42000 Hz), Mercury lamp (Philips 500-Watt E40 220-230V), and UV-Vis Spectrophotometer (Agilent Technologies-Cary 60) were used.

### 2.2. Preparation of ZnO Nanoparticles

The method used to synthesize ZnO nanoparticles in this study was used in the previous study conducted by Tong *et al.* [21]. The reference of the materials and the amount were based on the study conducted by Mandal *et al.* [26].

A total of 1.0975 g of Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O was dissolved in 60 mL of absolute methanol and stirred until

completely dissolved, labeled as Solution I. Meanwhile, 0.8417 g of KOH was dissolved in 30 mL of absolute methanol until it was completely dissolved, labeled as Solution II. Next, Solution II was added to the solution I gradually using the dropwise method and constantly stirred at room temperature. Stirring is carried out for 3 hours. The solution obtained was refluxed at 60°C for 3 hours using a water bath. After that, the samples were aged for 4 d, then centrifuged at 4,000 rpm for 20 min, and the pellets were taken and then washed with water several times. Furthermore, the ZnO nanoparticles were collected by filtering using a Buchner funnel and rinsed with water several times and rinsed with ethanol at the end. Finally, ZnO nanoparticles were obtained.

### 2.3. Photocatalytic Activity Experiment

10 mL of rhodamine B solution was inserted into the vial bottle, then 0.01 gram of ZnO nanoparticles was added, labeled, and then sonicated for 30 min (1800 s). The vial bottle containing rhodamine B solution was stored for 30 min in a dark room to investigate the effect of dark adsorption on the degradation of rhodamine B. Then, this vial bottle was exposed under visible light with the time varied for 30, 45, 60, 75, and 90 min, respectively. After that, it was centrifuged at 4,000 rpm for 20 min, and the supernatant was taken to test its absorbance using a UV-Vis spectrophotometer at 553 nm.

## 3. Results and Discussion

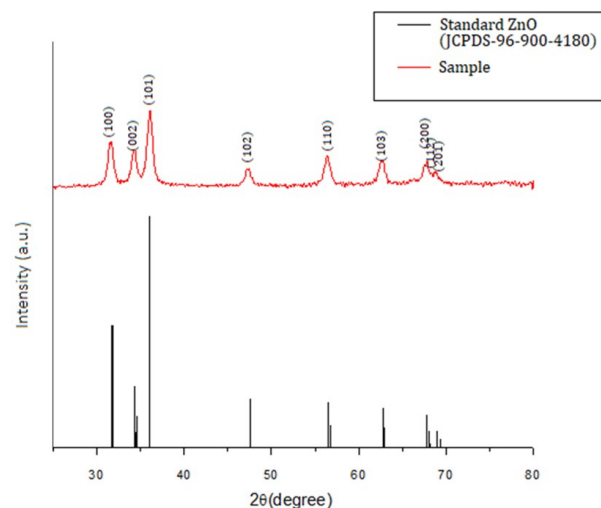
### 3.1. Characterization

An X-Ray Diffraction (XRD) analysis was carried out to determine the synthesized ZnO's crystal structure. It can be observed from **Figure 1** that the diffraction peaks are mainly located at 31,44°; 34,32°; 36,10°; 47,44°; 56,45°; 62,80°; 67,85°; 68,93° and 69,32° [JCPDS card no.96-900-4180] corresponding to (100), (002), (101), (102), (110), (103), (200), (112) and (201) crystal faces, respectively [27]. All the diffraction peaks present in the XRD pattern in **Figure 1** are strong and readily indexed as a hexagonal wurtzite ZnO crystal structure. That indicates that the ZnO that was successfully synthesized was highly crystallized [25]. Broad peaks indicate that ZnO synthesized consists of nano-sized particles [28]. No other peaks were detected, which means that there was no crystal phase or other compound, thus indicating the crystal purity of the synthesis. The peak with the highest intensity is found at  $2\theta = 36.10^\circ$  with the Miller index (101). This data can determine the mean size of the crystal using the Scherrer equation [18], as shown by equation (1).

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where D is the size of the crystals formed; k is the proportionality constant with a value of 0.9;  $\lambda$  is the wavelength of the CuK $\alpha$  radiation X-rays (1.54 Å or 0.154 nm);  $\beta$  is the width of the maximum diffraction peak or known as FWHM (Full Width at Half Maximum), and  $\theta$  is the Bragg angle.

The average crystallite size is estimated to be 22.77 nm. This relatively small crystal size indicates the success of the synthesis method in synthesizing nano-sized particles. Various studies with the same method, namely sol-gel, have been carried out, as shown in **Table 1**. The ZnO synthesized by the sol-gel method in this study is relatively small compared to other sol-gel methods caused by methanol as the solvent. Based on research conducted by [21], the resulting particle size is controlled by the type of solvent. Alcohol used as a solvent can form hydrogen bonds with the ZnO nucleus and inhibit the growth of the particles. It is said that the longer alkyl groups will weaken the negative charge of the oxygen atom, then its weaker hydrogen bond with the ZnO nucleus and consequently a weaker growth inhibition. The use of methanol solvent, which has the shortest alkyl group, is expected to inhibit growth more than other types of alcohol. Different solvents cause different particle sizes. Further particle size differences lead to differences in the bandgap.



**Figure 1.** XRD pattern of ZnO particles

To investigate the surface morphology of the synthesized ZnO particles, an analysis was carried out using SEM. The results of SEM analysis on ZnO synthesized by the sol-gel method are shown in **Figure 2**.



**Figure 2.** SEM images of ZnO particles

Table 1. Table of results of various studies using the sol-gel method.

Precursors	T (°C)	Structure	Application	Morphology	Size	Ref
Zn(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O, NaOH, PEG, HNO <sub>3</sub>	800	Hexagonal wurtzite	For electrode materials for supercapacitor applications.	Spherical shape, less homogeneous	50 nm (XRD) 50 - 70 nm (TEM)	[16]
Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O, extract of <i>Periconium sp.</i> , deionized water	700	Hexagonal wurtzite	Antimicrobials <i>Staphylococcus aureus</i> (Gram-positive), <i>Escherichia coli</i> (Gram-negative), and <i>Candida albicans</i> (fungi) as well as antioxidants	Spherical shape, homogeneous	40 nm (XRD) 16-78 nm (SEM)	[20]
Plant extracts of the <i>Geraniaceae</i> family, ZnCl <sub>2</sub> ·6H <sub>2</sub> O, NH <sub>4</sub> OH, distilled water	700	Hexagonal wurtzite	Antimicrobials (bacteria and fungi)	Spherical shape, homogeneous	5 -15 nm (TEM)	[17]
Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O, ethanol, glacial acetic acid, triethanolamine, distilled water	500	Hexagonal wurtzite	Photocatalyst to degrade methyl blue (MB)	granular, spherical, homogeneous structure	6,68-7,87 nm (XRD) 100 - 300 nm (SEM)	[18]
Zn(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O, ethanol, APTES, KOH	78	Hexagonal wurtzite	-	Spherical shape, homogeneous	2,85 and 3,68 nm (XRD) 3 nm (TEM)	[28]
Zn(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O, NaOH, distilled water-ethanol,	50-90	Hexagonal wurtzite	-	Hexagonal shape, homogeneous	16-32,2 nm (XRD) 10-35 nm (SEM)	[25]
Zn(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O, NaOH, distilled water	100	Hexagonal wurtzite	Nanocatalysts for biodiesel production	Hexagonal shape, homogeneous	44,37 nm (XRD)	[29]
Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O, PVA, distilled water, ethanol	500	Hexagonal wurtzite	Photocatalyst to degrade methyl blue (MB)	Semi-spherical shape, not homogeneous	22,3 nm (XRD)	[30]
Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O, <i>Hibiscus sabdariffa</i> extract, distilled water	400	Hexagonal wurtzite	Photocatalyst to degrade methyl blue (MB)	Spherical shape, not homogeneous	8,71-38,63 nm (XRD)	[19]
Zn(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O, ethanol, m-cresol	600	Hexagonal wurtzite	H <sub>2</sub> S gas sensor	Spherical shape, not homogeneous	34 nm (XRD)	[31]
Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O, gelatine, distilled water	500, 600 and 700	Hexagonal wurtzite	Dye adsorbent	Spherical shape, less homogeneous	15, 18, and 22 nm (XRD)	[32]

The SEM image of ZnO that has been successfully synthesized has the morphology of spherical particles. This is due to the solvent and the reaction rate of the mixing of the precursors. The dropwise method used when adding KOH to Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O is an effort to control the shape and growth of crystals. Previously, as reported [33], when the rate of addition of some OH<sup>-</sup> was carried out slowly, the growth of the ZnO crystal structure would be more uniform so that high homogeneity was achieved. Meanwhile, the reaction will run faster when the two precursor solutions of Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O and KOH are mixed directly and not dropwise. This fast reaction causes an excess of OH<sup>-</sup> in the solution so that the growth will move along the direction of the lattice plane [0001]. Thus, the structure formed is a nanosheet. The most exciting finding from this previous study is that when the addition of OH<sup>-</sup> is relatively slow, this will limit the growth towards the lattice plane [0001].

The SEM results above also show that ZnO is agglomerated but relatively homogeneous. Based on the results of measurements using the ImageJ application, the distribution of particle sizes and their distribution is obtained, as can be seen in Figure 3, with a particle size of about 22-24 nm. The size obtained is then compared with previous studies, as shown in Table 1. It shows that the sol-gel method used in this study can produce particles with relatively small sizes so that they qualify as nanoparticles.

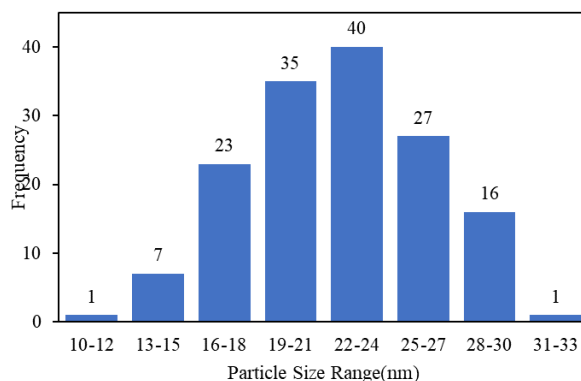


Figure 3. Histogram of the synthesized ZnO particle size distribution.

### 3.2. Photocatalytic Activity

After the product particles have been identified as ZnO compounds based on XRD and SEM characterization results. Further investigations are carried out for photocatalytic activity in degrading the widely used and complicated to degrade dyes, rhodamine B. The Sonication process is carried out for 30 min. ZnO is dispersed in solution. The surface area of ZnO interacts with Rhodamine B is maximal so that the degraded rhodamine B is maximal. In addition, if the ZnO particles precipitate, they will close together so that only part of it receives the energy from the exposed rays and is partially activated. A previous study conducted by Das and Bhattacharyya [34] stated that the amount of ZnO catalyst added to 1 g/L rhodamine B did not cause a significant change. In this study, there was no variation in the mass of ZnO. Based on the work reported by Dodoo-Arhin *et al.* [8], the degradation activity of rhodamine B with and without a catalyst and UV light was tested and it was concluded that neither adsorption nor photolysis

reactions occurred. Thus, a catalyst and light source were needed to degrade this dye compound in order more environmentally friendly. Besides, Rhodamine B and other dyes are difficult to degrade either without the help of a catalyst or without energy from light or photons. The degradation activity of rhodamine B without the help of a catalyst without the help of light and the degradation activity of rhodamine B with the help of a catalyst without light irradiation has negligible values.

In this study, Dodoo-Arhin *et al.* [8] synthesized nanoparticles using the sol-gel method by varying the calcination temperature (400, 500, and 600 °C) as a photocatalyst to degrade rhodamine B under UV exposure with optimum conditions of 0.2 g ZnO in 10 ppm rhodamine B resulted in efficient degradation of around 95.41%. By comparing with these studies, the ZnO produced from this study can be said to be competitive. The optimum point for the variation in irradiation time is 90 minutes because it has the most excellent efficiency, as shown in Figure 4. So, it can be concluded that the irradiation time is comparative to the degradation efficiency.

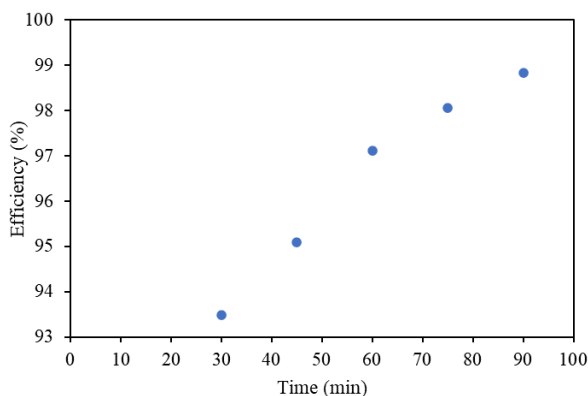


Figure 4. Effect irradiation time toward efficiency in degrading rhodamine B.

Heterogeneous photocatalysts generally follow the Langmuir-Hinshelwood kinetic model, which can be simplified to pseudo-first-order:

$$\ln \frac{C_i}{C_f} = kt \quad (2)$$

where  $C_i$  is the initial concentration and  $C_f$  is the final concentration. The photodegradation rate of rhodamine B using a ZnO photocatalyst was made by plotting the dye irradiation time (min) as the x-axis and  $\ln C_i/C_f$  as the y-axis, as shown in Figure 5. The photodegradation rate curve for rhodamine B is  $y = 0,0291x + 1,7895$ . As a result, the value of the photodegradation rate constant for rhodamine B using synthesized ZnO is  $0.0291 \text{ min}^{-1}$ .

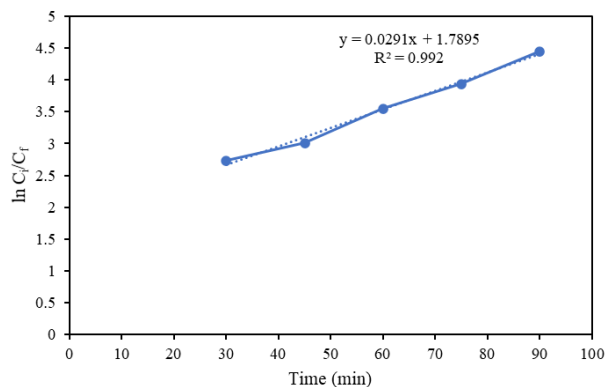


Figure 5. Photodegradation rate of rhodamine B using synthesized ZnO

The light used is visible light from the Mercury lamp (Philips 500-Watt E40 220-230V). At the time of exposure, the intermolecular reaction that occurs is shown in Figure 6. The light carrying  $h\nu$  energy will activate ZnO. When the amount of energy absorbed is sufficient or proportional to the Energy gap ( $E_g$ ), it will cause the excitation of electrons from the valence band (VB) to the conduction band (CB), causing the left band to form holes ( $h^+$ ). In contrast, the conduction band will be rich with electrons ( $e^-$ ). This  $E_g$  is the amount of energy required for an electron to excite. The holes will attract  $\text{OH}^-$  ions from the water and turn them into  $\cdot\text{OH}$ , while the electrons will change  $\text{O}_2$  to  $\cdot\text{O}_2^-$ , which will attract  $\text{H}^+$  ions from the water and turn into  $\cdot\text{OH}$  compounds. Hydroxide radical compounds ( $\cdot\text{OH}$ ) have an essential role in breaking down or degrading dyes to become simpler molecules such as  $\text{CO}_2$  and  $\text{H}_2\text{O}$  to be more environmentally friendly.

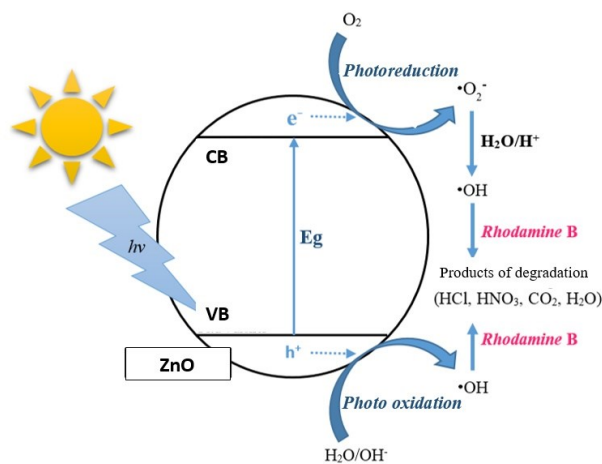
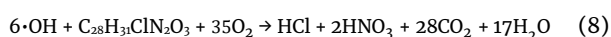
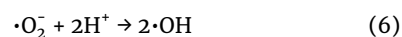
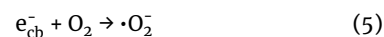
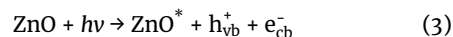


Figure 6. Photocatalytic mechanism of ZnO particles.

The chemical reactions that occur can be more precisely observed in the following reaction equations:



#### 4. Conclusion

From the research results, it can be concluded that ZnO nanoparticles were successfully synthesized by a simple sol-gel method by using reflux at 60°C and aging for four days, resulting in a white powder with a yield of 76.73%. XRD results showed that the ZnO nanoparticles were successfully synthesized and showed high purity with a 22.77 nm hexagonal wurtzite structure. The morphology of ZnO based on SEM analysis results shows that the ZnO synthesized form has a spherical particle shape and has good homogeneity with sizes ranging from 22–24 nm. The photocatalytic test results of ZnO nanoparticles in the degradation process of Rhodamine B showed excellent activity at the optimum time of 90 min with an efficiency of 98.83%.

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