

Analysis of Fe-doped ZnO thin films for degradation of rhodamine B, methylene blue, and Escherichia coli under visible light

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Analysis of Fe-doped ZnO thin films for degradation of rhodamine b, methylene blue, and *Escherichia coli* under visible lightHeri Sutanto^{1,2}, Ilham Alkian^{2,3}, Mukholit Mukholit¹, Arsyadio Aditya Nugraha¹, Eko Hidayanto¹, Indras Marhaendrajaya¹ and Priyono Priyono¹¹ Department of Physics, Faculty of Science and Mathematics, Diponegoro University, Jawa Tengah 50275, Indonesia² Smart Materials Research Center (SMARC), Diponegoro University, Jawa Tengah 50275, Indonesia³ Graduate Program of Environmental Science, School of Postgraduate Studies, Diponegoro University, Jawa Tengah 50241, IndonesiaE-mail: herisutanto@live.undip.ac.id**Keywords:** thin film, Fe-doped ZnO, photodegradation, rhodamine b, methylene blue, *Escherichia coli*

Abstract

ZnO is a popular photocatalyst that is often used for the degradation of dyes and bacteria. However, the catalytic performance of ZnO is only optimal under UV light exposure. This study aims to determine the degradation performance of rhodamine b, methylene blue, and *Escherichia coli* using 0, 5, 10, 15, and 20% Fe-doped ZnO (ZnO:Fe). Deposition of thin film was carried out using the sol-gel method with a spray-coating technique, while the degradation was carried out under halogen light exposure for 3 h. The optical characterization results show that 20% Fe-doped ZnO has the highest transmittance and the lowest energy band gap of 3.21 eV based on Tauc's plot method. All thin films are hydrophilic with the largest contact angle of 68.54° by 20% Fe-doped ZnO and the lowest contact angle of 52.96° by 5% Fe-doped ZnO. The surface morphology of the thin film resembles a creeping root that is cracked and agglomerated. XRD test results show that the thin film is dominated by ZnO peaks with a wurtzite structure with a hexagonal plane phase and a crystal size of 115.5 Å. The 20% Fe-doped ZnO thin film had the most efficient degradation performance of 70.79% for rhodamine b, 65.31% for blue, and 67% for *E. coli* bacteria. Therefore, Fe-doped ZnO is a brilliant photocatalyst material that can degrade various pollutants even under visible light.

1. Introduction

Water pollution by dyes is still a big problem that has not been resolved until now [1–5]. Most of the pollution is caused by the industrial waste of clothing, fabrics, and jeans. Dyestuff waste directly discharged into the environment without being processed first becomes a source of pollution and can cause various hazards, such as toxic effects and reduced light penetration ability in waters [6, 7]. Among the various textile dyes, the most widely used are methylene blue and rhodamine b [8, 9]. In addition to dyes, water pollution by *Escherichia coli* bacteria is also a problem [10, 11]. Recently, there have been 'viral' cases of health problems caused by *Escherichia coli* contamination such as fever, typhoid, and diarrhoea experienced by residents in several districts in Indonesia, such as Banjarnegara, Gunung Kidul, and Klaten. Based on the investigation results, it was found that the water used by the community in the area contained *Escherichia coli* bacteria that exceeded the safe threshold as water that humans can consume.

The presence of dye compounds and *Escherichia coli* bacteria is a serious problem that needs proper handling. Photodegradation is one of the methods to decompose industrial waste dyes. Degradation by utilizing the aid of light offers a relatively inexpensive solution and is easy to implement in Indonesia. In operation, the principle used in this method is to activate photocatalyst materials from semiconductor materials, such as CdS, Fe₂O₃, ZnO, TiO₂, Bi₂O₃, Cu₂O, WO₃ etc [12–16]. Semiconductor materials that are easily found and most often used are TiO₂ and ZnO [17–19]. ZnO is an excellent oxidizing agent used as a photocatalyst and has a higher efficiency due to the strong absorption of UV from the solar spectrum. Compared to other catalysts, ZnO

is suitable for the detoxification process of colour waste in water because it produces H_2O_2 more efficiently [18, 20].

The photocatalytic process occurs when ZnO material in water is exposed to UV light, electrons in the valence band will be excited to the conduction band. This process produces electrons (e^-) in the conduction band and holes (h^+) in the valence band. Then electrons react with oxygen molecules from water to form superoxide anion radicals ($O_2^{\cdot-}$), and holes will react with hydroxyl ions from water to form hydroxyl radical compounds (OH^{\cdot}). Superoxide reacts with electrons, and H^+ ions from water form H_2O_2 compounds then react again with electrons to produce hydroxyl radicals [21]. Electrons and holes generate hydroxyl radical compounds then oxidize pollutant molecules (M) to produce degraded compounds (M'). If M is a dye such as rhodamine b and methylene blue, M' is dyes with a more straightforward carbon chain and lower concentration. If M is *Escherichia coli*, M' is *Escherichia coli* with less coloni.

In fact, in the actual environment, UV levels in sunlight are microscopic. Therefore, the optical properties of ZnO need to be improved to produce electrons with visible light energy. To improve the properties of ZnO, doping with metal ions is the most effective way to produce structural changes. The optical band gap energy and ferromagnetic properties can be controlled by doping the micro conductor with a transition metal such as Fe. Fe-doped ZnO nanoparticles play an essential role in the photodegradation of organic pollutants [22] and exhibit better photocatalytic activity than undoped ZnO nanoparticles [23]. Several researchers have prepared Fe-doped ZnO nanoparticles, but high doping amounts, control of optical properties to enhance photocatalytic activity, and degradation of multianalyte are rare. In our previous study, the degradation of pollutants by Fe-doped ZnO was still using a UV excitation source [7]. In this study, the degradation was carried out under halogen rays as a representation of actual sunlight. The synthesis results were then characterized and tested for photodegradation of methylene blue, rhodamine b, and *Escherichia coli*.

2. Materials and methods

2.1. Materials

The materials used in this study were methylene blue ($C_{16}H_{18}ClN_3S$), rhodamine b ($C_{28}H_{31}N_2O_3Cl$), glass preparation, Iron Nitrate ($Fe(NO_3)_3 \cdot 9H_2O$, Merck KGaA 99.95%), Zinc Acetate Dehydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$, Merck KGaA 97%), Acetone (Merck KGaA 99.5%), Isopropanol (Merck KGaA 99.5%), Methanol (Merck KGaA 99.9%), Aquades, Monoethanolamine (Merck KGaA), while the tools used are Mini Compressor (Krisbow, AS 186), VMC digital balance (VB 304), Degradation lamp (20 W), Hotplate Stirrer (Yellow MAG HS 7), and spray (Krisbow HS, 1200333).

2.2. Method

The Fe-doped ZnO sol-gel preparation was prepared by preparing 0.5 M zinc solution from 3.046 g $Zn(CH_3COO)_2 \cdot 2H_2O$ and 26 ml isopropanol stirred with a magnetic stirrer temperature of $60^\circ C$ for 15 min until homogeneous [24]. The solution was stirred with a magnetic stirrer at $60^\circ C$ for 15 min until homogeneous. Monoethanolamine (MEA) was dropped into the solution and stirred on a hotplate at $60^\circ C$ for 15 min until the solution was colourless or transparent. Fe Nitrate with various concentrations of 0, 5, 10, 15, and 20% was mixed and then stirred for 15 min at a temperature of $60^\circ C$. Thin film deposition was carried out using a spray-coating technique, and previously the glass substrate was cleaned using the Radio Corporation of America (RCA) method. The resulting solution is sprayed for 30 min at a temperature of $450^\circ C$. The degradation test was carried out by making a solution of 10 ppm dye (rhodamine b and methylene blue). Photodegradation was carried out under visible light for 180 min with observations every 30 min. The degradation test of *Escherichia coli* bacteria was carried out with actual samples from the Rasamala river under visible light for 180 min.

2.3. Characterization

The morphology and chemical composition of the compounds in the thin film was observed using the Analytical Scanning Electron Microscopy—Energy Dispersive x-ray instrument (SEM—EDX JEOL JSM-6510LA). Analysis of the thin film crystallinity using x-ray Diffraction instrument (Shimadzu Maxima XRD-700). Determination of the contact angle of the tips film using a digital microscope endoscope camera instrument. Optical characteristics and efficiency photodegradation of dyes and *Escherichia coli* were analyzed based on the results of the UV-vis Spectrophotometer (Shimadzu UV-vis 1240 SA).

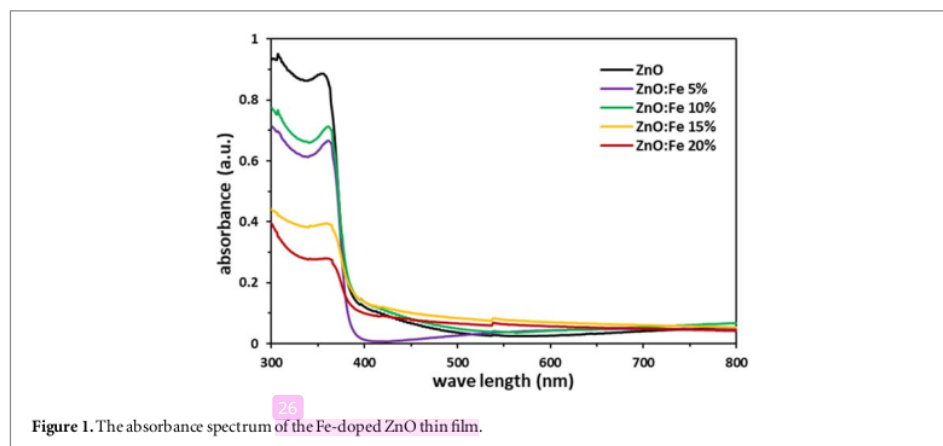


Figure 1. The absorbance spectrum of the Fe-doped ZnO thin film.

Table 1. Fe-doped ZnO thin film contact angle against rhodamine b and methylene blue.

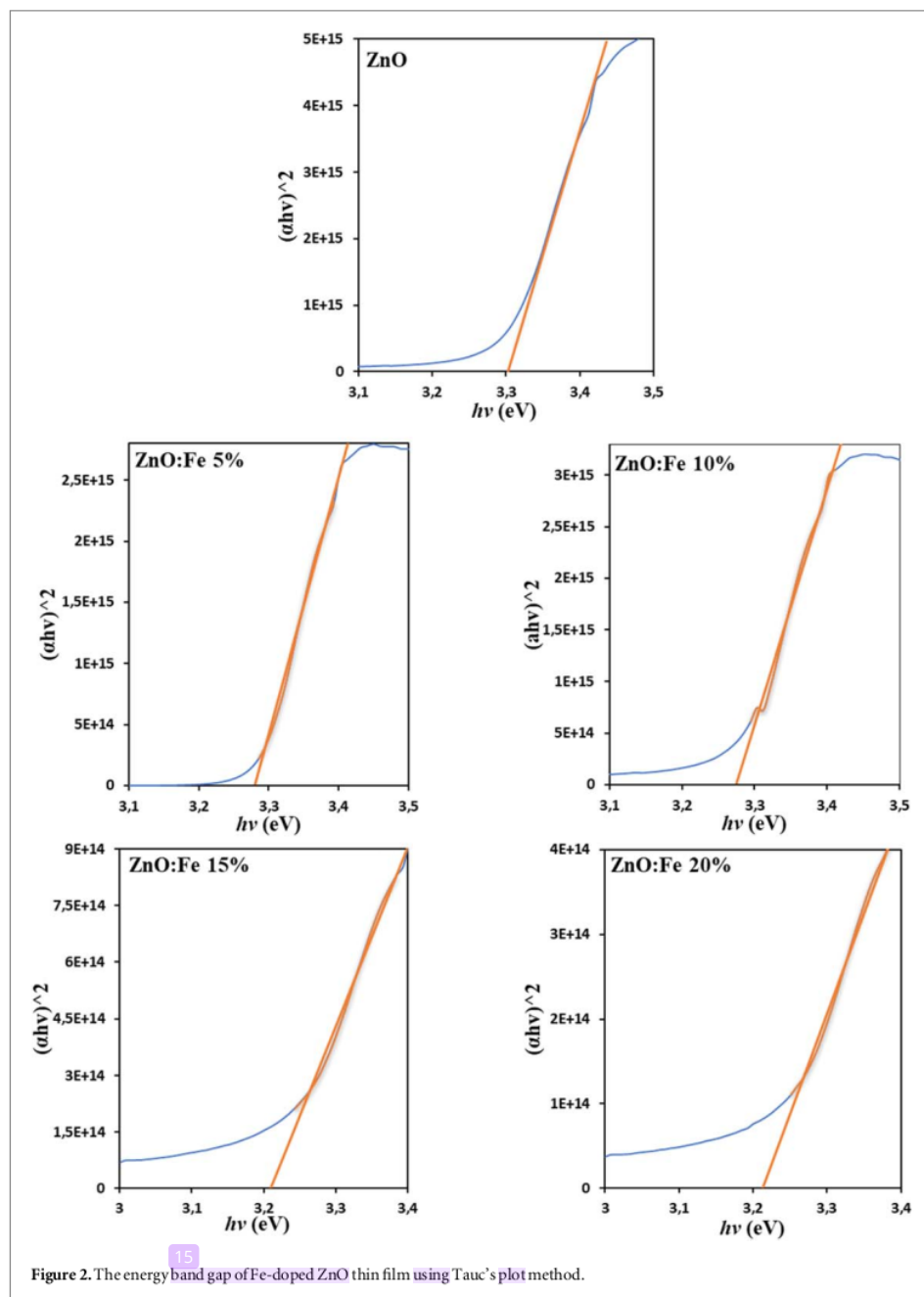
Thin film	Contact angle (°)	
	Rhodamin b	Methylene blue
Undoped ZnO	62.53	63.27
5% Fe-doped ZnO	52.96	57.62
10% Fe-doped ZnO	55.55	61.81
15% Fe-doped ZnO	65.75	62.83
20% Fe-doped ZnO	66.89	68.54

3. Results and discussion

The optical properties of the Fe-doped ZnO thin film obtained from the UV-vis test are shown by transmittance and absorbance graphs. Tests of the absorbance spectrum of Fe-doped ZnO thin film in the wavelength range of 300–800 nm are shown in figure 1. The highest transmittance is by 20% Fe-doped ZnO thin film at the same wavelength, while the highest absorbance is undoped ZnO. Substitution of Zn atoms by Fe atoms will form close distances between particles with less light [25]. It indicates that the concentration of Fe is very influential with the transmittance value because it can produce a different number of electron-hole pairs for each sample. The less Fe, the fewer electrons are produced so that the photon energy absorbed by the electrons to be also excited less and the transmittance value is increased.

The energy band gap value is processed using Tauc's plot method as shown in figure 2. In this study, the shift that occurs is redshift, as shown from the results of 0%–20% Fe-doped ZnO, namely 3.32, 3.28, 3.25, 3.21, and 3.21 eV. This result is consistent with the study reported by Ariyakkani *et al* (2017) for Fe-doped ZnO thin films, where the Eg value decreased from 3.38 eV for undoped ZnO to 3.07 eV for 20% Fe-doped ZnO [26]. A decrease in the energy bandgap indicates the shift that occurs as the concentration of Fe increases. Thus, the decrease in the energy bandgap occurs because the valence electrons of Fe larger than Zn will cause Fe to become a donor atom near the conduction band so that the electron transition requires less energy to the conduction band [27, 28]. The decrease in the energy band gap value can also be explained by the presence of Fe atoms in ZnO can suppress crystal growth. In addition, the inclusion of Fe in ZnO also causes defects in ZnO crystals that cause high light absorption, which is very necessary to increase the photoactivity of visible light from ZnO [29].

The hydrophilicity test was carried out between the dye solution and the surface of the film that had been coated on a glass plate using a Contact Anglemeter. Table 1 shows all the Fe-doped ZnO films forming a contact angle of less than 90° which means the films can interact well with water. Despite being dropped with two different dyes (rhodamine b and methylene blue), the resulting contact angle increased uniformly as the Fe concentration in ZnO increased, from 62.53° to 66.89° and 63.27° to 68.54°. This result is in contrast to ZnO doped with other materials, such as La, Na, Pb, and Mg, where the addition of a doping concentration of less than 7.5% reduces the contact angle value [30, 31], including our previous study where the addition of Fe also lowers the value of the contact angle [7].



Interestingly, these results indicate that the addition of Fe doping less than 10% will increase the hydrophilic properties, while the addition of Fe doping more than 10% will increase the hydrophobic properties. This change in properties is probably due to differences in the microstructure and morphology of each doped film. Changes in properties that lead to an increase in hydrophobic properties will be advantageous for degradation applications because it increases the surface area and causes an increase in analyte adsorption on the catalyst surface [32]. The higher the contact angle formed by the thin film, the higher the degradation efficiency produced under halogen light.

SEM characterization is needed to determine the material's physical properties, including its microstructure and surface morphology. Figure 3 shows the results of Fe-doped ZnO thin film characterization using SEM with

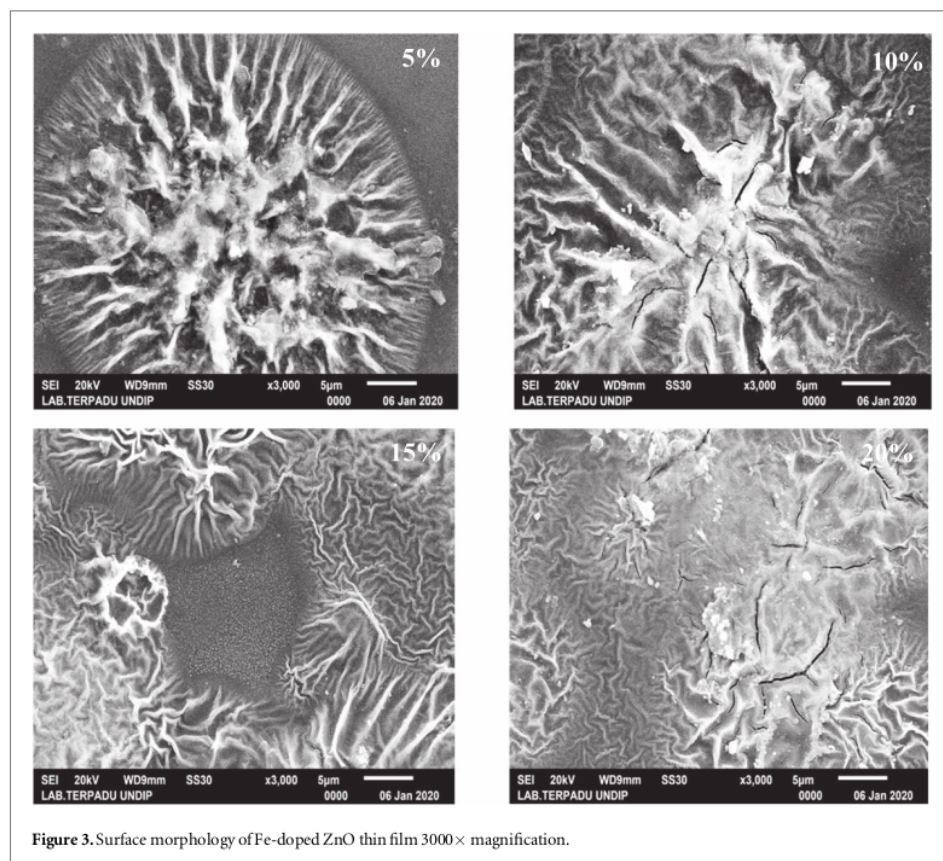


Figure 3. Surface morphology of Fe-doped ZnO thin film 3000 \times magnification.

3000 \times magnification. Fe-doped ZnO thin film appears to have a root-like morphology grouped and evenly distributed over the entire substrate surface. The root-like surface morphology is caused by the bonds between the particles deposited in the presence of high temperatures on the substrate. These bonds result in the grains becoming fused and forming a structure that looks like a root [33]. The diameter of the size formed ranged from 150 nm to 500 nm with an average particle size of 333.75 nm. A high concentration of Fe dopant resulted in the appearance of agglomeration and aggregation in ZnO morphology [34], also causing irregular cracked surfaces.

Characterization using EDX is needed to determine the chemical properties, including the material's elemental composition. Table 2 shows that O, Fe, and Zn atoms were successfully formed on the substrate. The increase in the percentage of mass and Fe atoms and the increase in the number of concentrations. It indicates an increase in the molecules that make up the thin film formed. It causes the resulting energy band gap to become smaller, and the ability of Fe-doped ZnO to photodegrade in the visible light spectrum is getting better. X-ray diffraction test results were carried out to determine the phase contained in the sample. From the results of the XRD test, analysis was carried out by matching the spectrum of the XRD test results with JCPDS data (Joint Committee on Powder Diffraction Standard) which helps know what compounds are contained in the Fe-doped ZnO. JCPDS data used are JCPDS 36-1451 (ZnO), JCPDS 06-0696 (Fe), and JCPDS 24-0072 (Fe₂O₃). Figure 4 shows the XRD test results for 20% Fe-doped ZnO showing several diffraction peaks with the most dominant peak belonging to ZnO with 2θ used between 20°–90° and the wavelength used = 1.5405 Å.

The XRD test results showed that the ZnO sample had a wurtzite structure with hexagonal plane phases showed that the diffraction peaks were at $2\theta = 31.76^\circ, 34.26^\circ, 36.10^\circ, 47.48^\circ, 56.54^\circ, 62.90^\circ$, and 67.82° with Miller indices (100), (002), (101), (102), (110), (103), and (200) [35–37]. This result is similar with the study reported by Roguai and Djelloul (2021), where all the peaks of the XRD spectrum (100), (002), (101), (102), (110), (103), and (200) for Fe-doped ZnO (0%–10%) are identified as single-phase ZnO wurtzite structure with the space group $P6_3mc$ [38]. During synthesis, the doping addition of ferrite nitrate led to a peak at 24.22° , corresponding to (012) Fe₂O₃ [39]. In the hexagonal structure, lattice constants (a and c), unit cell volume (V), crystal size (D), lattice strain (ϵ), and atomic packing fraction (APF) are calculated using the following formula [40]:

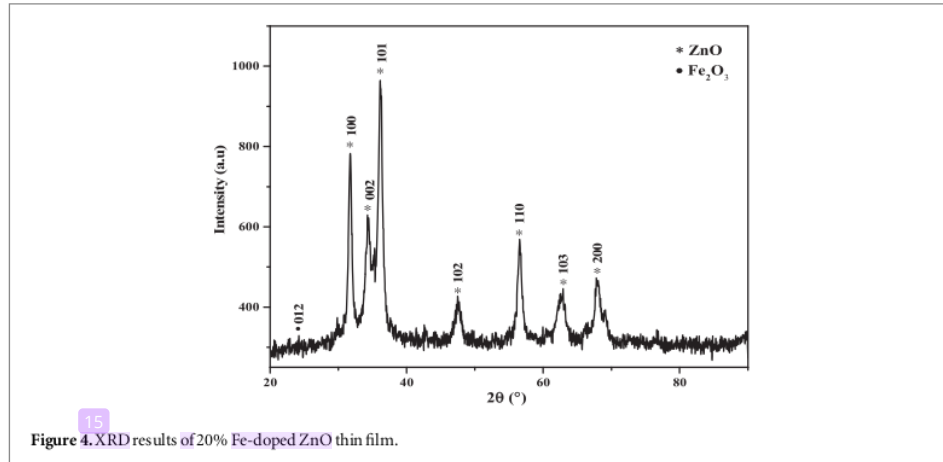


Figure 4. XRD results of 20% Fe-doped ZnO thin film.

Table 2. The mass composition of the chemical elements Fe-doped ZnO thin film.

Element	Mass of Fe-doped ZnO (%)			
	5%	10%	15%	20%
O	49.03	54.96	58.38	53.31
Fe	2.05	3.41	3.58	5.47
Zn	48.92	41.63	38.04	41.21

Table 3. Crystal parameters of Fe-doped ZnO.

Parameter	Value	Unit
a	1.509586	Å
c	1.57997	Å
V	3.118137	Å ³
APF	1.154747	%

$$a = \frac{\lambda}{\sqrt{3} \sin \theta_{(100)}} \quad (1)$$

$$c = \frac{\lambda}{\sqrt{3} \sin \theta_{(002)}} \quad (2)$$

$$V = \frac{\sqrt{3}}{2} a^2 c \quad (3)$$

$$APF = \frac{2\pi}{3\sqrt{3}} \frac{a}{c} \quad (4)$$

The results of calculations using the formulas (1) to (5) are shown in table 3 below:

X-ray diffraction is also used to determine the Crystal Size (D) by using the Debye Scherrer equation approach as follows:

$$D = \frac{K \lambda}{\beta \cos \theta} \quad (5)$$

Where λ is the wavelength of x-rays (1.54056) and β is the value of Full Width at Half Maximum (FWHM), and K is the crystal form factor whose values are between 0.9. The Debye-Scherrer equation is then modified into equation (6) as follows:

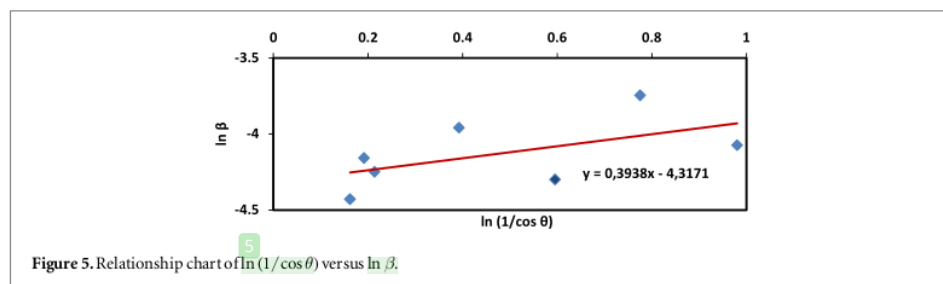


Figure 5. Relationship chart of $\ln(1/\cos \theta)$ versus $\ln \beta$.

Table 4. Calculation of $\ln(1/\cos \theta)$ and $\ln \beta$.

2θ	Crystal plane orientation	$1/\cos \theta$	β (rad)	$\ln 1/\cos \theta$	$\ln \beta$
31.76	100	1.1758	0.01193	0.16195	-4.42894425
34.26	002	1.21092	0.01564	0.19138	-4.1582305
36.10	101	1.23844	0.01427	0.21385	-4.24943083
47.48	102	1.4805	0.01908	0.39238	-3.95921498
56.54	110	1.8134	0.01358	0.59521	-4.29944439
62.90	103	2.17099	0.02362	0.77518	-3.74586958
67.82	200	2.66511	0.017	0.98025	-4.07475961

$$\ln \beta = \ln \frac{K \lambda}{D} + \ln \frac{1}{\cos \theta} \quad (6)$$

The calculation of the values $\ln(1/\cos)$ and \ln XRD analysis results in all crystal plane orientations are shown in figure 5 and table 4:

The value of intercept from the graph of the relationship $\ln(1/\cos)$ as the x-axis and \ln as the y axis is equal to $((K)/D)$, so equation (6) can be modified into the following equation:

$$D = \frac{K \lambda}{\exp(y_0)} \quad (7)$$

Where y_0 is the intercept value of the graph $\ln(1/\cos)$ versus \ln , the intercept value from the graph above is -4.3171, so the crystal size is 115.5 Å. This result may be the smallest crystal size among other samples not tested, considering that several previous studies increasing the Fe content reduced the lattice parameters and the average crystal size [34, 41, 42].

Figure 6 and table 5 show the results of the photodegradation carried out to test the photocatalyst activity of Fe-doped ZnO. Photocatalyst activity was tested to degrade rhodamine b, methylene blue, and *Escherichia coli* under halogen to represent visible light. Photocatalyst reactions that occur in the reaction of Fe-doped ZnO material with visible light can excite electrons and leave holes [43, 44]. The electrons interact with the oxygen present in the water to produce superoxide. Meanwhile, hydroxyl radicals are generated from the interaction between holes and water. Hydroxyl radicals are potent oxidizing agents, so they are capable of oxidizing organic compounds. The radicals formed will break down the dye molecules to produce H_2O and CO_2 [45].

Photodegradation of the thin film under visible light for rhodamine b and methylene blue degradation based on 5%–20% Fe-doped ZnO, are 64.53; 65.57; 67.36; 70.79% and 60.64; 62.53; 62.34; 65.31%. The photodegradation of *Escherichia coli* was 25, 42, 50, and 67%. The most optimal degradation ability is possessed by a 20% Fe-doped ZnO, both on rhodamine b, methylene blue, and *Escherichia coli*. The energy band gap value influences the effectiveness of the photocatalyst thin film performance. The small energy band gap indicates that the energy required to excite electrons from the valence band to the conduction band is getting smaller. With the same amount of energy, a thin film with a small energy band gap can produce more electron-hole pairs than a thin film with a large energy band gap. Therefore, the smaller the energy band gap, the higher the degradation ability under visible light. On the other hand, the larger the energy band gap, the lower the degradation ability under visible light.

The presence of Fe found in EDX measurements is believed to form a trapping state between the conduction band and the valence band, which can inhibit the recombination rate of electron and hole pairs and increase photocatalytic activity. This result was confirmed by Bousslama *et al* (2017), who investigated Fe-doped ZnO for the degradation of rhodamine b [28], Saleh and Djaja (2014) for the degradation of methyl orange [46], and

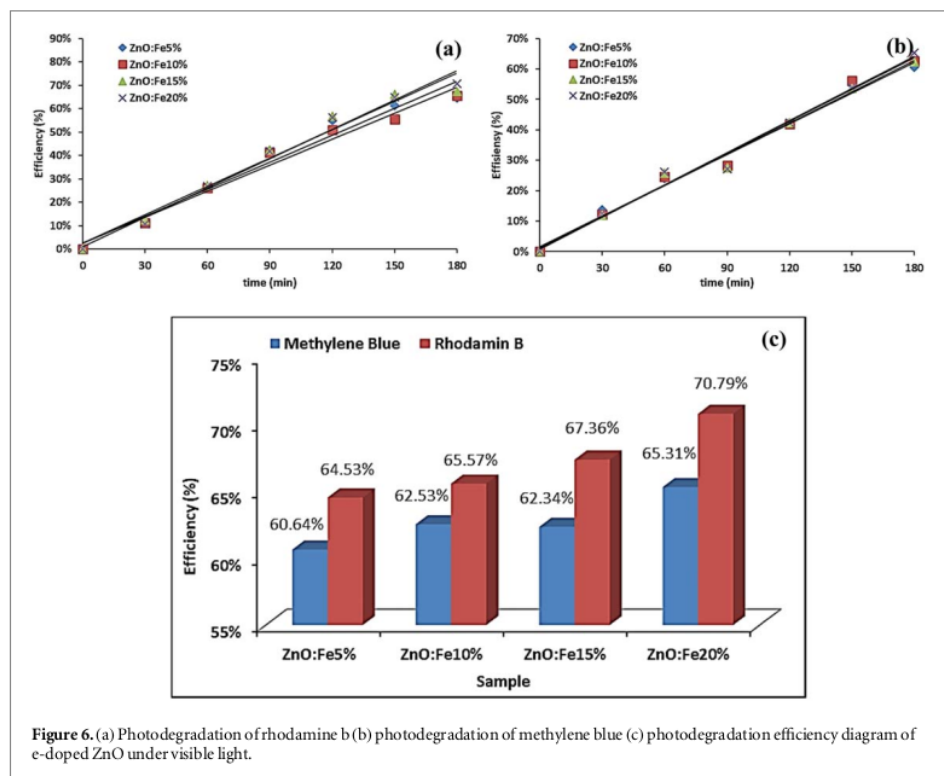


Figure 6. (a) Photodegradation of rhodamine b (b) photodegradation of methylene blue (c) photodegradation efficiency diagram of e-doped ZnO under visible light.

Table 5. Bacterial degradation test results.

Thin film	Degradation efficiency (%)
5% Fe-doped ZnO	25
10% Fe-doped ZnO	42
15% Fe-doped ZnO	50
20% Fe-doped ZnO	67

Roguai and Djelloul (2021) for the degradation of methylene blue [38]. Fe^{2+} and Fe^{3+} ions obtained are known to form 2 new energy levels in the energy gap. One energy level is above the valence band, which refers to the 3d orbital of Fe^{3+} , and the other is below the conduction band because the electron energy level of the Fe^{2+} ion is lower than the 3d Zn in the conduction band [25, 46].

When halogen light is given, electrons can be excited from Fe^{3+} and the valence band to the conduction band. After the electrons are excited, Fe^{3+} will be converted to Fe^{4+} , which can interact with hydroxyl ions and produce OH hydroxyl radicals. Simultaneously, electrons excited to the valence band can react with oxygen and produce superoxide radicals ($\text{O}_2^{\cdot-}$). The formed active radical species will initiate a series of redox processes to break down dye compounds into simpler ones and cause *Escherichia coli* cell membranes to lyse. Therefore, developing a thin film with appropriate optical properties such as 20% Fe-doped ZnO would be advantageous for the degradation of liquid pollutants since electron-hole pairs can be generated only by a visible light excitation source.

4. Conclusions

The increase in Fe dopant impacts improving optical properties, decreasing the energy bandgap and increasing the photocatalytic properties of ZnO thin films. A Fe-doped ZnO thin film has a root-like surface morphology dominated by ZnO peaks with a wurtzite structure with a hexagonal plane. Fe dopant determines the hydrophilicity of film, and the magnitude of the contact angle is directly proportional to the degradation

efficiency. 20% Fe-doped ZnO is the best photocatalyst thin film formula for degrading dyes and *Escherichia coli* under visible light.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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