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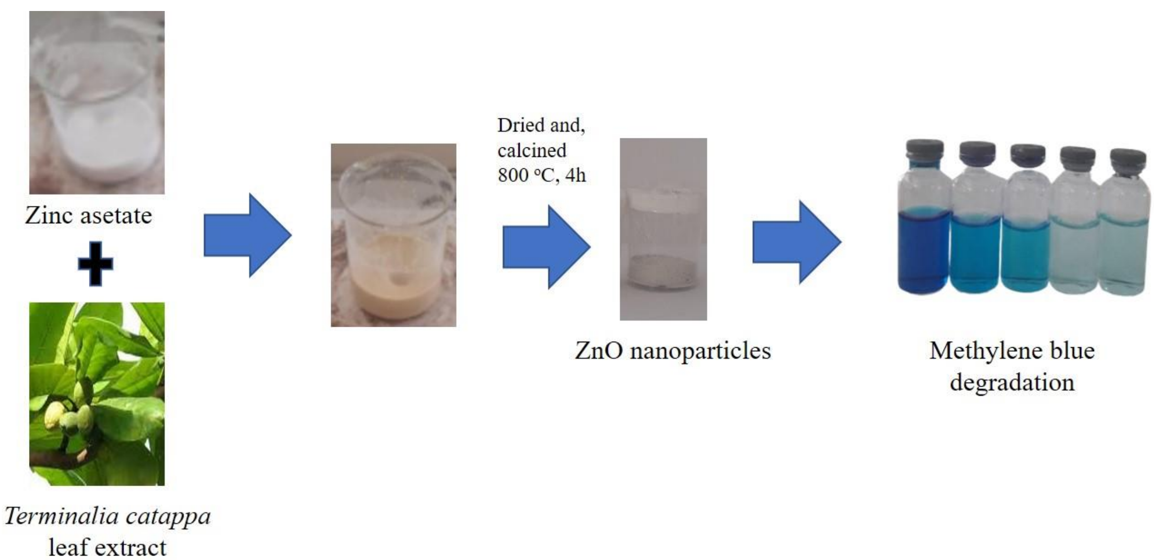
GREEN SYNTHESIS OF ZNO NANOPARTICLES FROM LEAF EXTRACT OF *Terminalia catappa* AND ITS APPLICATION AS PHOTOCATALYST

Mei Rizda Brilianti[1], Amaria[2], Amalia Putri Purnamasari[2], Arikasuci Fitonna Ridassepri[2], and Dina Kartika Maharani[2,*]
[1]Department of Chemistry, Universitas Negeri Surabaya, Jl. Ketintang, Gayungan 60231, Surabaya, East Java, Indonesia
[2] Department of Chemistry, Universitas Negeri Surabaya, Jl. Ketintang, Gayungan 60231, Surabaya, East Java, Indonesia

*Correspondence: dinakartika@unesa.ac.id

ABSTRACT
ZnO nanoparticles were successfully synthesized using a green synthesis method with ketapang leaf extract as a reducing and stabilizing agent. The objectives of this study were to synthesize ZnO nanoparticles using ketapang leaf extract, characterize the ZnO nanoparticles, and evaluate their photocatalytic activity for the degradation of methylene blue. The ZnO nanoparticles were synthesized using the sol-gel method. Characterization was conducted using Fourier Transform Infrared Spectroscopy (FT-IR) to identify functional groups and X-ray Diffraction (XRD) to determine phase and crystal size. FT-IR characterization revealed the presence of Zn-O at a wavelength of 450 cm^{-1} and -OH groups at 3406 cm^{-1}. XRD characterization indicated a ZnO crystal size of 17.5 nm. The photocatalytic activity of ZnO nanoparticles from ketapang leaf extract showed that an optimal ZnO nanoparticle mass of 30 mg achieved a degradation rate of 81.92% for methylene blue. The optimal contact time was 80 minutes, resulting in a degradation of 81.56%. The optimal methylene blue concentration was 5 ppm, with a degradation rate of 87.91%.

Keywords: green synthesis, ZnO nanoparticles, catappa leaves,.



INTRODUCTION
Among other metal oxide nanoparticles, ZnO nanoparticles are preferred due to their non-toxic properties, which provide high mobility and good thermal stability[1]. ZnO nanoparticles are n-type inorganic semiconductors with a hexagonal phase and wurtzite structure, featuring a wide band gap of 3.37 eV[2]. Due to their high exciton binding energy, ZnO exhibits strong ultraviolet (UV) emission of 60

MeV[3]. The advantages of using ZnO include its abundant availability in nature, resulting in low cost and easy synthesis[4].

ZnO nanoparticles can be synthesized using physical or chemical methods. Physical synthesis does not involve reactions and merely reduces bulk materials to nanometer size or mixes very small materials, like clusters, into smaller particles without altering their properties. In chemical synthesis, precursors are used to produce new materials[5]. Both physical and chemical synthesis methods have drawbacks, including the need for expensive equipment, high-temperature processes, and the use of environmentally harmful chemicals[6].

Green synthesis of nanoparticles aims to reduce or eliminate the use of hazardous chemicals. The goal of green synthesis is to achieve desired results without producing harmful by-products[7]. Green synthesis is a cost-effective and non-toxic method that uses microorganisms, biological materials, biomolecules, and plant extracts as stabilizers, thereby reducing hazardous waste[8].

ZnO nanoparticles synthesized using plant extracts and their derivatives are more stable compared to those synthesized using other organisms[9]. Various plant extracts have been used in the synthesis of ZnO nanoparticles, including kingkilaban stem extract[10], daphne leaf extract[11], and pineapple fruit extract[12]. Plant parts such as sap, stems, seeds, fruits, flowers, leaves, and roots are rich in phytochemical compounds that can reduce metal salts to metal oxides at the nanoscale[13]. This is due to the presence of proteins and secondary metabolites in plant extracts, which act as reducing agents, stabilizers, and capping agents in the biosynthesis of ZnO nanoparticles[14,15]. The mechanisms of stabilization and prevention of ZnO nanoparticle agglomeration involve creating steric barriers, controlling particle growth by reducing surface energy to prevent aggregation, and forming stable coordination compounds with Zn(II) metal ions[16].

Previous studies employed *Terminalia catappa* extracts for the green synthesis of ZnO nanoparticles. However, nearly all of those studies focused on the material's antibacterial properties. To the best of our knowledge, no publication has yet explored the photocatalytic performance of *Terminalia catappa* mediated ZnO nanoparticles for the degradation of methylene blue. By addressing this gap, our work demonstrates the potency of *Terminalia catappa* derived ZnO as a photocatalyst for methylene blue dye. In this study, ketapang leaf extract (*Terminalia catappa*) was used as a reducing and stabilizing agent in the green synthesis of ZnO nanoparticles. The resulting ZnO nanoparticles will be characterized and tested for their photocatalytic activity against methylene blue dye.

MATERIAL AND METODS

Materials

Zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$) (Merck), NaOH (Merck), distilled water, and methylene blue. Ketapang leaves (*Terminalia catappa*), were obtained from Surabaya (Indonesia). The leaves were air dried and blended for further use.

Methods

Extraction of ketapang leaves

A beaker containing 100 grams of Ketapang leaf powder is added with 1000 mL of 70% ethanol solvent and tightly sealed with aluminum foil to protect it from sunlight. The maceration process is carried out for two days, with occasional stirring. After two days, the mixture of Ketapang leaf powder and 70% ethanol solvent is filtered to obtain the macerate. The macerate is then left to stand overnight, and the filtrate is separated from the residue. The Ketapang leaf extract is then concentrated using a rotary evaporator at 60°C[17].

Synthesis of Zinc oxide nanoparticle (ZnO)

A total of 25 mL of zinc acetate dihydrate solution is added with 20 mL of Ketapang leaf aqueous extract, followed by the dropwise addition of NaOH. The mixture is then stirred at room temperature for 2 hours until it reaches a pH of 12. The solution is then filtered to obtain the precipitate. The precipitate is dried in an oven at 150°C and calcined at 800°C for 4 hours[18].

Characterization of ZnO with FTIR

ZnO nanoparticles in powder form will be characterized using FT-IR to identify organic and organometallic compounds. Functional group analysis will follow the ASTM D-6348 (2010) standard with the following steps: the ZnO solid is prepared with KBr to form a pellet, then analyzed using FT-IR at a wavelength range of 400-4000 cm^{-1}. The vibrational peaks that appear and the functional groups acting as reducing or stabilizing agents will be observed. The obtained data will show vibrational peaks indicating the presence of functional groups in the compound. Based on the observed functional groups, the atomic bonds in the sample being measured can be identified[19].

Characterization of ZnO with XRD

ZnO nanoparticles in powder form will be characterized using X-ray diffraction (XRD) to identify the phases and crystal sizes of the ZnO nanoparticles. A 1 gram sample of the powder is placed in a diffractometer. In the diffractometer, the sample is bombarded with an electron beam, resulting in diffraction patterns that are translated into an X-ray intensity spectrum against 2θ, with values ranging from 10° to 100°. The crystal phase can be determined by comparing the data with the provided JCPDS database. Crystal size can be calculated using computer simulation from the X-ray diffraction pattern or from the Full Width at Half Maximum (FWHM) of the diffraction peaks using the Debye-Scherrer equation:

$$d = \frac{k.\lambda}{\beta.cos\theta}$$

where d is the crystal size, θ is the Bragg angle, K is the Scherrer constant (K = 0.9), λ is the X-ray wavelength, and β is the Full Width at Half Maximum (FWHM).

Determination of the Standard Wavelength for Methylene Blue

Methylene blue with a concentration of 3 ppm is measured for its wavelength using a UV-Vis spectrophotometer in the range of 200-800 nm. The maximum wavelength is determined from the highest absorbance value.

Calibration Curve for Standard Methylene Blue Solution

A 100 ppm methylene blue solution is pipetted in amounts of 0.5, 1, 1.5, 2, and 2.5 mL and placed into a 50 mL volumetric flask. The solution is then diluted to the mark and homogenized to obtain concentrations of 1 to 5 ppm

Photocatalytic Activity Test of ZnO Nanoparticles

Photocatalytic Test with Mass Variation

The test is performed under UV light using a UV Pro instrument (6 watts, 220 volts). The mass variation test is conducted by placing 20 mL of 10 ppm methylene blue into separate beakers. ZnO nanoparticles in varying masses of 10 mg, 20 mg, 30 mg, and 40 mg are added to each beaker as adsorbent mass. The solution is stirred using a magnetic stirrer for 10 minutes[20]. After 10 minutes, the samples are filtered using Whatman No. 1 filter paper, and the resulting solution is tested using UV-Vis spectrophotometry

Photocatalytic Test with Exposure Time Variation

The photocatalytic activity test with exposure time variation is conducted by placing 20 mL of 10 ppm methylene blue into separate beakers. Then, 20 mg of ZnO nanoparticle powder is added to each beaker. The exposure times are varied for each adsorbent mass at 20, 40, 60, 80, and 100 minutes using a magnetic stirrer. The solution is then analyzed using a UV-Vis spectrophotometer to determine the effect of each sample on methylene blue[21]. The percentage of methylene blue degradation is calculated using the following formula:

$$\textit{Persen degradation} = \frac{Co - C}{C0} \times 100\%$$

$$\textit{Persen degradation} = \frac{A0 - C}{A0} \times 100\%$$

Where C_0 is the initial concentration of methylene blue, and C is the final concentration of methylene blue. Similarly, A_0 represents the initial absorbance, and A is the absorbance of methylene blue after the reaction[22].

The photocatalytic degradation test with methylene blue concentration variation is conducted by placing methylene blue solutions of 5, 10, 15, and 20 ppm into separate beakers, each containing 20 mL. The optimum mass (mg) of ZnO nanoparticle powder is added to each beaker. The contact time is set to the optimum time (minutes), and the solution is stirred using a magnetic stirrer. The solution is then analyzed using a UV-Vis spectrophotometer to determine the effect of each sample on the methylene blue[22].

RESULTS AND DISCUSSION

Results of the synthesis of ZnO nanoparticles with Ketapang Leaf Extract

The green synthesis of nanoparticles in this study uses a bottom-up approach, where the reactions involve reduction or oxidation processes[19]. In this research, zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$) was used as a precursor dissolved in distilled water. Then, 20 mL of ketapang leaf extract was added and heated for 15 minutes at 95°C while stirring. The heating and stirring aimed to accelerate the reaction for forming ZnO nanoparticles. Continuous stirring during synthesis can influence the rate of nanoparticle formation and prevent agglomeration, allowing for uniform distribution of the nanoparticles[23]. The temperature of 95°C was chosen because, according to Matufu, et al[24], at this temperature, antioxidant compounds and secondary metabolites can be optimally utilized without damaging functional groups.

According to Fabiani et al[25], nanoparticle formation occurs due to the reduction process of secondary metabolites, specifically phenolic compounds such as flavonoids and tannins. These secondary metabolites act as reducing agents undergoing oxidation, while Zn serves as the oxidizing agent undergoing reduction. The ketapang leaf extract also acts as a stabilizer, wrapping (capping) the formed ZnO nanoparticles to prevent agglomeration, ensuring the stability of the resulting nanoparticles. The oxidation reaction of the secondary metabolites (polyphenols) generates electrons that donate their electrons to convert Zn^{2+} ions into ZnO[26].

The synthesis of ZnO nanoparticles begins with the formation of $Zn(OH)_2$ in a basic solution at pH 12, indicated by the production of a transparent yellowish solution during the reaction. A pH of 12 is used because, between pH 6 and 11, unstable ZnO nanoparticles would formed[27]. When Zn^{2+} and OH^- reach the critical solubility threshold (the hydrolysis stage), a colloid is produced, marked by a pale yellow color change. This color change is an indicator of nanoparticle formation, occurring due to the redox reaction between Zn^{2+} metal ions and the *Terminalia catappa* leaf extract[28]. The Zn^{2+} cation then reacts with hydroxide anions to form the stable complex $Zn(OH)_4{}^{2-}$ (the polymerization stage). With the presence of H_2O and energy from continuous stirring using a magnetic stirrer, the $Zn(OH)_4{}^{2-}$ complex can dissociate to form Zn^{2+} and OH^- ions, which subsequently form ZnO nanoparticles (the transformation stage). Extracts of *Terminalia catappa* are particularly attractive because they contain abundant flavonoids, amino acids, alkaloids, and other phenolic compounds that can favour the reduction and caps for the formation of well stabilised ZnO nanoparticles.

The formation of ZnO nanoparticles is also achieved through a calcination process at a temperature of 800°C. The purpose of calcination is to oxidize zinc nanoparticles into ZnO[20]. The temperature of 800°C is based on previous studies conducted by Rahman[20] and Saba[29], which successfully synthesized ZnO nanoparticles. Zinc is a highly reactive element, making it easy to form ZnO during heating[20]. The heating occurs at a specific temperature with the presence of oxygen gas, allowing the metal species to oxidize easily into their metal oxide form[20]. The resulting ZnO nanoparticles appear as a powder with a grayish-white color.

Figure 1. ZnO Nanoparticles

To confirm the formation of ZnO nanoparticles and their potential application as photocatalysts, characterization was performed using FT-IR and XRD techniques.

Characterization results using FT-IR Spectroscopy

The results of the green synthesis of ZnO with various volumes of ketapang leaf extract were analyzed using Fourier Transform Infrared (FTIR) spectroscopy to identify the functional groups in the samples. The identified functional groups of the obtained ZnO nanoparticles are illustrated in Figure 2.

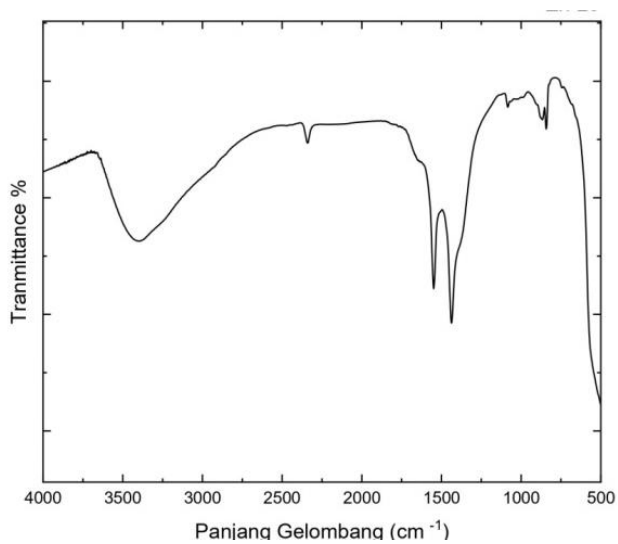


Figure 2. FTIR Spectrum of ZnO Nanoparticles

In the spectrum produced by the ZnO nanoparticles, an absorption peak at 3419.33 cm^{-1} corresponds to the O-H stretching vibration[30]. According to Wolkers et al[31], the wavelength range of 1500-1200 cm^{-1} represents the bending vibrations of C-H from functional groups CH, CH_2, and CH_3, indicating that the absorption peak at 1436.16 cm^{-1} corresponds to C-H bending vibrations. Additionally, the absorption peaks at 430.66 cm^{-1}, 425.00 cm^{-1}, 481.00 cm^{-1}, and 450.00 cm^{-1} correspond to the Zn-O stretching vibrations, in line with the findings of Shathy, et al[32]. This result aligns with prior research that reported the synthesis of ZnO nanoparticles using extracts of *Terminalia catappa*.

Characterization results using XRD

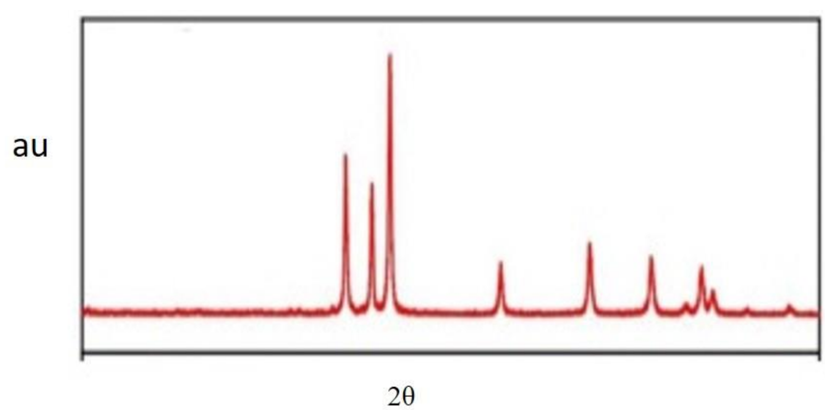


Figure 3. Diffraction pattern of ZnO nanoparticles

The diffraction pattern of ZnO nanoparticles is depicted in Figure 3. XRD analysis showed sharp and narrow diffraction peaks at scattering angles (2θ) of 31.75°, 34.37°, 36.24°, 47.48°, 56.56°, 62.95°, 66.19°, 67.83°, and 68.99°, representing the hexagonal wurtzite phase of ZnO and confirming the formation of ZnO nanoparticles with high crystallinity. The observed diffraction peaks closely matched the ZnO peaks from the standard JCPDS No. 36-1451[33].No additional diffraction peaks indicating impurities were detected, suggesting that the synthesized nanoparticles possess high purity. The crystallite size of ZnO nanoparticles was 17,6 nm. This result was in accordance with previous research of ZnO nanoparticles from Ananas comosus peel extract which obtained crystallite size 17,02 nm [34].

ZnO Nanoparticles as Photocatalysts

The determination of the maximum wavelength of methylene blue is essential because each solution exhibits different wavelengths. This condition represents the point at which a substance shows the highest absorption. Measuring at the same wavelength enhances measurement sensitivity, leading to more accurate analysis and reduced analytical errors. The maximum wavelength obtained for methylene blue is 663 nm. The standard curve for methylene blue, shown in Figure 5, was created by measuring the absorbance of methylene blue at concentrations ranging from 2 to 10 ppm at 663 nm.

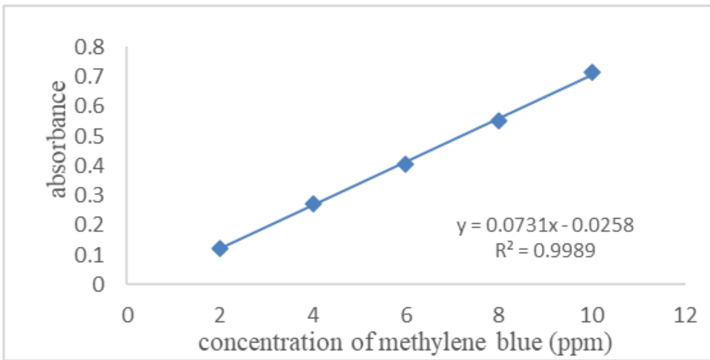


Figure 4. Methylene Blue Standard Curve

From Figure 4, it is evident that concentration is directly proportional to absorbance. The linear regression equation obtained is y=0.0731x−0.0258y = 0.0731x - 0.0258y=0.0731x−0.0258 with a coefficient of determination (R^2) of 0.9989. This result indicates a strong correlation, as the coefficient of determination is close to 1, suggesting that the model fits the data well. The maximum wavelength (λ) of methylene blue was 663 nm which obtained from the maximum absorbance of methylene blue at concentration of 3 ppm using a UV-Vis spectrophotometer.

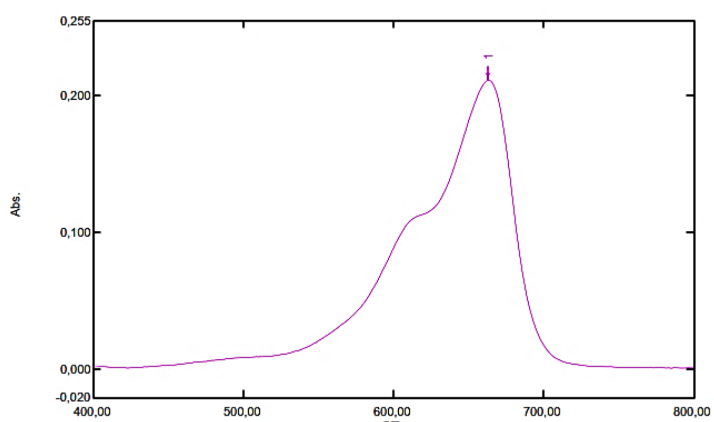


Figure 5. Methylene Blue Maximum Wavelength Graph

Application of ZnO nanoparticles with mass variations

The highest methylene blue photodegradation result was 81.90% with a ZnO nanoparticle mass of 30 mg and a UV irradiation time of 80 minutes. Photodegradation refers to the breakdown of organic compounds, such as methylene blue, into harmless and simpler substances like water and carbon dioxide using photocatalysts and UV light.

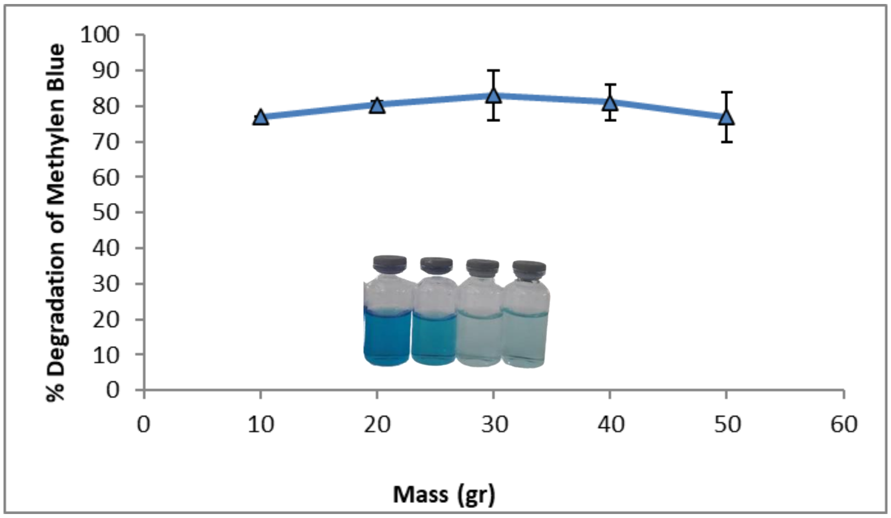


Figure 6. Results of Photodegradation of Methylene Blue with Variations in Photocatalyst Mass

Based on Figure 5, the percentage of degradation using varying catalyst masses of 10 mg, 20 mg, 30 mg, and 40 mg were 75.00%, 78.55%, 81.92%, and 79.43%, respectively. The lowest degradation percentage was observed with a catalyst mass of 10 mg at 75.00%, while the highest degradation percentage was achieved with 30 mg of catalyst at 81.92%. As shown in Figure 6, there was an increase in methylene blue photodegradation activity from 10 mg to 30 mg, with less significant results at 40 mg.

Among the four variations of ZnO nanoparticle mass used, 30 mg was determined to be the optimum mass, where photocatalytic performance was maximized. The increase in methylene blue degradation percentage with increasing catalyst mass is attributed to the growing number of active sites on the photocatalyst. This leads to more photon energy being absorbed, resulting in an increased formation of hydroxyl radicals (OH•) that react with methylene blue in solution. Therefore, as more hydroxyl radicals are produced, a greater amount of methylene blue can be degraded[35].

However, at a mass of 40 mg, there was a decrease in photodegradation activity due to reaching a saturation point. The mass of the photocatalyst can significantly influence the efficiency of methylene blue degradation in solution. When the photocatalyst mass exceeds the saturation point, it tends to form aggregates that can cover the active sites on the photocatalyst surface[36]. Additionally, excessive photocatalyst mass can cause the solution to become very turbid, limiting the absorption of UV light and potentially scattering it, leading to reduced photon capture[37]. Conversely, if the photocatalyst mass is too low, there may not be enough ZnO nanoparticles to effectively bind methylene blue in solution.

Application of ZnO nanoparticles with time variation

The testing began with weighing 30 mg of ZnO nanoparticles for each variation. This experiment utilized different contact times of methylene blue solution with ZnO nanoparticles for 20, 40, 60, 80, and

100 minutes. A 10 ppm methylene blue solution of 20 mL was mixed with 30 mg of ZnO nanoparticles, and then stirred for the specified contact times of 20, 40, 60, 80, and 100 minutes.

After stirring, the solution was filtered using filter paper to separate the methylene blue solution from the used ZnO catalyst. The filtered methylene blue solution was then analyzed for absorbance using a UV-Vis spectrophotometer at a wavelength of 663 nm. The absorbance values obtained from the four different contact time variations were used to calculate the concentration of methylene blue after treatment. These absorbance values were then processed into a graph, which is displayed in Figure 7.

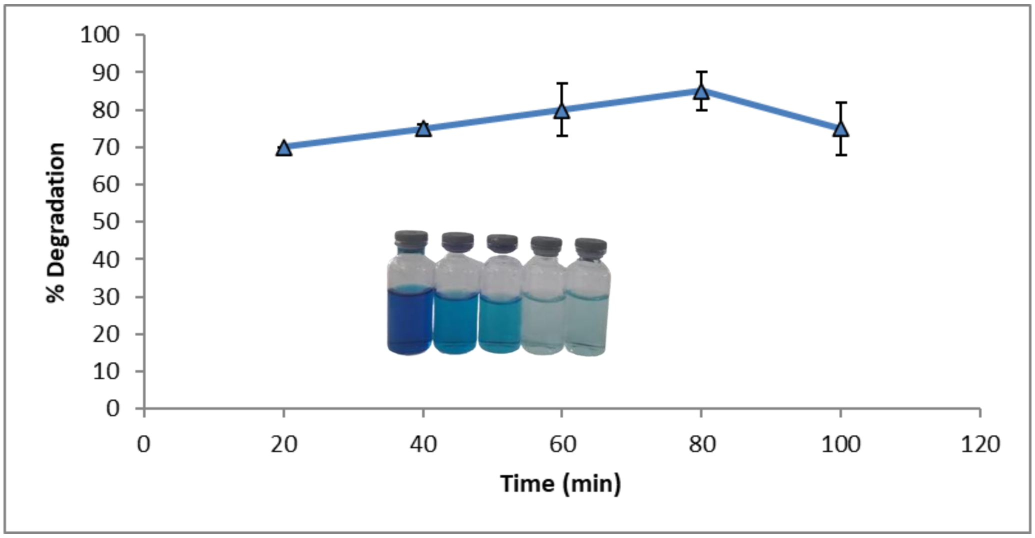


Figure 7. Results of Photodegradation of Methylene Blue with Varying Photocatalyst Time

CONCLUSION

ZnO nanoparticles have been successfully synthesized using a green synthesis method with ketapang leaf extract as a reducing and stabilizing agent. The resulting crystal size was 17.5 nm. The highest photodegradation of methylene blue achieved in this study was 81.92%, occurring with 30 mg of ZnO nanoparticles, a methylene blue concentration of 10 ppm, and UV exposure for 80 minutes.

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CONFLICT OF INTERESTS

The authors declare no conflict of interests related to the publication of this paper.

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