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Utilization of Natural Zeolite Impregnated with Fe for Decolorization of Methylene Blue

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Abstract

Wastewater containing methylene blue, discharged into rivers, significantly impacts water quality due to its resistance to natural degradation. This study investigated the treatment of methylene blue using the photo-Fenton method, employing UV light to generate hydroxyl radicals (•OH) through the reaction of hydrogen peroxide (H_2O_2) and Fe catalyst. Natural zeolite was used as a support material, activated with NaOH solution, and impregnated with $FeSO_4 \cdot 7H_2O$. Material characterization was conducted using SEM-EDS and XRD. Semi-quantitative EDS analysis indicated an iron content of 6.2 wt%. The XRD result shows that the crystalline iron phase was hematite. The photo-Fenton experiments were performed at a catalyst dosage of 0.1 g/L to degrade methylene blue with an initial concentration of 20 mg/L by varying pH levels (3, 5, 7) and H_2O_2 concentrations (15, 30, 45 mM). The optimal conditions were found to be a combination of 45 mM H_2O_2 concentration, pH 3, and under 365 nm UV lamp irradiation, achieving a maximum decolorization efficiency of 99.77% at 120 minutes. H_2O_2 concentration did not significantly affect final decolorization percentage, indicating that excess H_2O_2 does not enhance degradation beyond a certain threshold. The lowest final methylene blue concentration achieved was 0.05 mg/L, and the final chemical oxygen demand (COD) was reduced to 243.6 mg/L.

Keywords: natural zeolite, heterogeneous catalyst, photo-Fenton method, methylene blue

INTRODUCTION

The rapid development of industrialization has led to various environmental problems, one of the most pressing being water pollution (Dharmraj Khairnar et al., 2018). A significant contributor to this problem is the discharge of untreated wastewater from the textile industry. Based on data from the Ministry of Industry regarding the number of textile factories in Indonesia, at least over 25,000 registered textile companies are producing a wide range of textile commodities (Kementerian Perindustrian Republik Indonesia, 2018). These industries produce wastewater containing synthetic dyes that consist of non-biodegradable organic compounds. When released directly into the environment without adequate treatment, it can severely generate environmental pollution (Latupeirissa et al., 2018). The presence of dyes in water bodies affects the pH of water, thereby causing disruption and aquatic animals (Fransina & Latupeirissa, 2016).

Methylene blue, a synthetic dye known for stability and vibrant color, is one of the dyes commonly used in the textile industry. Besides disrupting the aquatic ecosystem, methylene blue also poses significant health risks. At certain concentrations, it exhibits toxicity that can cause increased heart rate, vomiting, and shock in humans (Anindika, 2019). According to the Minister of Environment Regulation number Kep51/MENLH/10/1995 regarding the quality standards for industrial liquid waste, the maximum allowable concentration for methylene blue in wastewater is 5-10 mg/L.

One promising alternative for treating synthetic dyes-contaminated wastewater is the photo-Fenton method (Vorontsov, 2019). The photo-Fenton method is an advanced oxidation process (AOP) that degrades organic compounds in wastewater using highly reactive hydroxyl radicals (•OH) (Kusumawati et al., 2025). These radicals are generated through the reaction of hydrogen peroxide (H_2O_2) with ferrous ions (Fe^{2+}), assisted by UV light to enhance •OH production.

Even though the application of homogeneous photo-Fenton shows high efficiency in degrading dyes, it suffers from limitations. The use of dissolved iron as a catalyst will require a post-treatment separation process to remove excess iron from the treated water and thus increase operational costs. To overcome this

limitation, the heterogeneous photo-Fenton has been explored. This approach uses solid-phase catalysts, which eliminate the need for iron removal after treatment (Hassan & Hameed, 2020).

Iron oxides have been investigated as heterogeneous Fenton catalysts. In contrast to a homogeneous system, it results in lower activity due to limited mass transfer and slow diffusion of H_2O_2 to the surface, which prevents the production of hydroxyl radicals (Quynh et al., 2021). Therefore, impregnating the iron catalyst on a solid support is one method to enhance performance. Because of its large surface area, ion exchange capacity, adsorption capacity, high thermal stability, and environmental friendliness, zeolite is a material that can be used as support (Hassan & Zahidi, 2019). The use of natural zeolite as a photocatalyst support has been reported by Agustina et al. (2020) and Sarabyar et al. (2024) with specific surface areas of 26.19 m^2/g and 12.24 m^2/g, respectively.

In the catalytic oxidation of organic pollutants, zeolite containing iron has demonstrated strong performance, providing both stability of the iron catalyst and efficient pollution degradation. According to Jayanudin & Kustiningsih (2018), phenol was effectively degraded by natural zeolite as a catalyst support of Fe catalyst in the photo-Fenton process, especially when the catalyst mass was raised. Using zeolite 4A as a catalyst support, Ikhlaq et al. (2019) discovered that the maximum decolorization of safranin was accomplished at pH 3, highlighting the crucial role that pH level plays in the photo-Fenton process. Additionally, using zeolite A as a catalyst support, Quynh et al. (2021) showed that the addition of H_2O_2 improved the decolorization of methylene blue, achieving a decolorization percentage of up to 93%.

Because it is inexpensive and widely available, natural zeolite has an advantage over synthetic zeolite in the heterogeneous photo-Fenton process (Jayanudin & Kustiningsih, 2018). However, activation is necessary to remove impurity material (Fabiani et al., 2017), and impregnation is necessary to increase the iron content of natural zeolite. In this study, the resulting iron-impregnated natural zeolite (natural zeolite-Fe) was applied as a catalyst in the photo-Fenton process to decolorize methylene blue. The effect of operating conditions such as pH and H_2O_2 concentration was studied to optimize the decolorization percentage.

METHODOLOGY

Materials and Instrumentals

The materials used in this research were natural zeolite from Indonesia, H_2O_2, $FeSO_4.7H_2O$, NaOH, HCl, and Methylene Blue. All chemical materials are pro analysis from Merck.

The instruments used in this research were NOVA Touch LX4 (USA) to identify surface area of sample with degassing temperature of 300°C (before analysis), SEM-EDS to identify the morphology of sample, Bruker D2 Pasher (Germany) to identify the crystal phases of sample, Genesys spectrophotometer (Thermo Fisher Scientific, USA) to identify concentration of methylene blue in solution.

Methods

Preparation and Activation of Natural Zeolite

The procedure for the preparation and activation of natural zeolite was adapted from as Ates (2018). To prepare the natural zeolite, its size was reduced, and it was sieved until it was smaller than 200 mesh. After that, the natural zeolite was cleaned with distilled water, dried for four hours at 120°C, then heated for four hours at 500°C. The chemical activation was performed in a three-neck flask by mixing natural zeolite with a 1 M NaOH solution. The solid-to-liquid ratio was 1:20 (g/mL). The mixture was then stirred and heated at a temperature of 90°C for one hour. After that, the activated natural zeolite was filtered and washed with distilled water until the pH of the filtrate was 7 and dried. The pH of filtrate was measured by a pH meter.

Impregnation of Natural Zeolite with Fe

The wet impregnation procedure referred to the studies of Hassan & Hameed (2020). Based on Hassan & Hameed (2020), Ikhlaq et al. (2019), and Rivas et al. (2019), the range of iron content was 3-10 wt%. A target loading of approximately 6 wt% was selected for this research. To prepare the material, 3.5 grams of $FeSO_4.7H_2O$ were dissolved in 20 ml of deionized water and then mixed with 10 grams of activated natural zeolite without adjusting pH. At 70°C, the mixture was heated and stirred until all the water evaporated. The solid was dried for twenty-four hours at 105°C, followed by calcination in air at 550°C for four hours. The calcination step was performed to promote oxidation of Fe species to Fe_2O_3. The theoretical Fe loading and potential Fe loss are described below.

Theoretical Fe loading

$$m_{Fe,\ added} = 3{,}5\ \text{gram} \times \frac{55.85}{278.06} = 0.703\ \text{gram}$$

$$w_{Fe,\ theoretical} = \frac{m_{Fe,\ added}}{m_{zeolite} + m_{Fe,\ added}} \times 100\%$$

$$w_{Fe,\ theoretical} = \frac{0.703\ gram}{10\ gram + 0.703\ gram} \times 100\% = 6.57\%$$

Measured Fe loading by EDX

$$w_{Fe,\ measured} = 6.2\%$$

Fe loss during processing

$$\text{Fe loss (\%)} = \frac{w_{Fe,\ theoretical} - w_{Fe,\ measured}}{w_{Fe,\ theoretical}} \times 100\%$$

$$\text{Fe loss (\%)} = \frac{6.57 - 6.2}{6.57} \times 100\% = 5.63\%$$

Characterization of Natural Zeolite and Zeolite-Fe

The Brunauer-Emmett-Teller (BET) method was used to determine the surface area of natural zeolite and activated natural zeolite. Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDS) was used to analyze morphology and identify the components contained in the zeolite, and X-Ray Diffractometer (XRD) was used to examine the crystal phase of natural zeolite-Fe.

Preparation of Methylene Blue Solution

The artificial wastewater containing methylene blue was prepared. A 1000 mg/L stock solution was made by dissolving 1 gram of methylene blue into 1 L of distilled water. Using dilution techniques, a concentration of 20 mg/L was attained. Each experiment used 500 mL of methylene blue solution with a methylene blue concentration of 20 mg/L (Yudha et al., 2024).

Heterogeneous Photo-Fenton Process

Effect of pH

As many as 0.05 grams of zeolite-Fe were mixed with 500 mL of methylene blue solution (pH 3). A solution of H_2O_2 with a concentration of 30 mM was added to the mixture. The reaction time started when the H_2O_2 solution was first added, and the UV lamp was turned on. Eight samplings were conducted at 5, 10, 15, 30, 45, 60, 90, and 120 minutes. The visible spectrophotometer was used to measure the methylene blue concentration. For the pH variation, tests were conducted at pH 3, 5, and 7.

Effect of H_2O_2 Concentration

As many as 0.05 grams of zeolite-Fe were mixed with 500 mL of methylene blue solution (pH 3). A solution of H_2O_2 with a concentration of 30 mM was added to the mixture. The reaction time started when the H_2O_2 solution was first added, and the UV lamp was turned on. Eight samplings were conducted at 5, 10, 15, 30, 45, 60, 90, and 120 minutes. The visible spectrophotometer was used to measure the methylene blue concentration. For variation of H_2O_2 concentration, tests were conducted at 15, 30, dan 45 mM.

Analysis

Color Analysis

Color analysis can be performed using a visible spectrophotometer with standard solutions of 0, 1, 2, 3, 4, and 5 ppm. The concentration of methylene blue before and after photo-Fenton was analyzed at a wavelength of 660 nm.

COD Analysis

The samples analyzed were samples before and after the photo-Fenton process that resulted in the highest decolorization percentage. A blank was prepared by mixing 2.5 ml of distilled water, 1.5 ml of dichromate reagent, and 3.5 ml of sulfate reagent in the Hach tube. On the other hand, 2.5 ml of the sample was placed into the Hach tube, and then dichromate and sulfate reagents were added in the same amount as in the blank. Once the digester reached a temperature of 150°C, the samples in the Hach tubes were inserted and processed for 120 minutes. After that, the tubes were cooled to room temperature. The samples were then transferred to an Erlenmeyer flask for titration by adding ferroin indicator. Titration was performed using 0.1 N FAS until the color changed to reddish-brown.

Data Analysis

Decolorization percentage was calculated with the following equation.

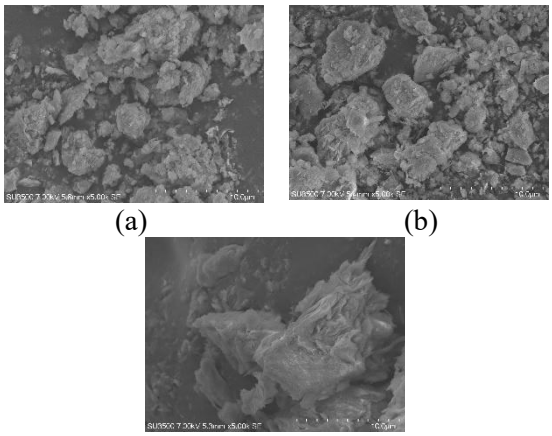
$$\boldsymbol{decolorization}\ (\%) = \frac{C_{in} - C_{out}}{C_{in}} \times \mathbf{100}\% \qquad (1)$$

C_{in} : initial concentration of methylene blue (mg/L)
C_{out} : concentration of methylene blue after photo-Fenton process at a certain time (mg/L)

RESULTS AND DISCUSSION

Preparation and Activation of Natural Zeolite

Characterization using SEM-EDS aims to determine the morphology of natural zeolite and activated natural zeolite. The morphology of natural zeolite and activated natural zeolite is shown in Figure 1.



(a) (b)

(c)

Figure 1. Morphology of (a) Natural Zeolite (b) Thermally Activated Natural Zeolite (c) Chemically Activated Natural Zeolite

Natural zeolite is thermally activated at a temperature of 500°C and chemically activated with a 1 M NaOH solution. Thermal activation led to the release of water molecules (H_2O) from within the natural zeolite's pore structure (Ngapa, 2017). This dehydration process enhances pore accessibility and increases the specific surface area, from 11.31 m^2/g to 29.32 m^2/g. As shown in Figure 1. (a) and Figure 1. (b), there are no significant changes to the zeolite structure when heated at 500°C, which aligns with the findings of Cadar et al. (2020). Additionally, EDS analysis results in Table 1 indicate a decrease in oxygen content, further supporting the loss of water during thermal activation.

The activation process with a 1 N NaOH solution breaks the siloxane (Si-O-Si) bonds within the natural zeolite framework through a process known as desilication. This results in the dissolution of silica (Gea et al., 2020), forming sodium silicate and generating mesopores. The reaction can be represented as follows.

$$2NaOH + SiO_2 \rightarrow Na_2SiO_3 + H_2O \quad (2)$$

In addition to desilication, this activation facilitates ion exchange, wherein certain cations within the natural zeolite framework are removed and replaced by Na^+ ions. This process brings the natural zeolite structure closer to a homoionic form. The Figure 1. (c) shows the morphology of particles after activation with NaOH. Furthermore, the EDS analysis result presented in Table 1 indicates a decrease in the Si element and an increase in the Na element content, supporting the occurrence of desilication and ion exchange.

Table 1. EDS Analysis Results of Natural Zeolite and Activated Natural Zeolite

Element	Percentage (%)		
	Natural zeolite	Natural zeolite after thermal activation	Natural zeolite after thermal and chemical activation
O	54.2	53.49	53.01
Na	2.45	0.1	1.77
Al	7.49	7.51	7.5
Si	31.67	35.56	32.34
K	1.7	1.58	1.55
Ca	2.49	1.76	3.83

Impregnation of Natural Zeolite with Fe

Morphology of natural zeolite impregnated with Fe (natural zeolite-Fe) is shown in Figure 2.

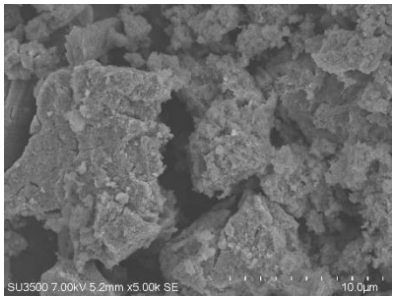


Figure 2. Morphology of Natural Zeolite Impregnated with Fe

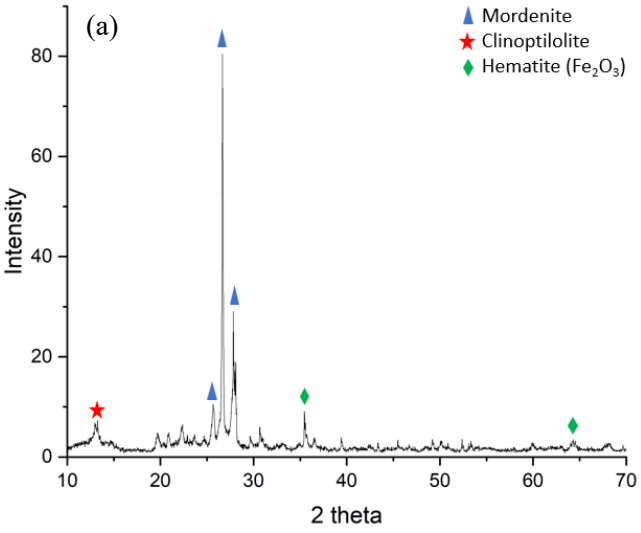
The impregnation process is carried out to increase the Fe content in natural zeolite. As shown in Figure 2, the morphology of natural zeolite-Fe appears coarser. This indicates the presence of Fe impregnation (19). According to the study by Kanari et al. (2018), $FeSO_4$ begins to decompose into Fe_2O_3 at around 500°C, as described by the following reaction (3). Fe_2O_3 is a semiconductor that can absorb UV light and react with H_2O_2 to produce hydroxyl radicals (Tia Deka, 2019).

$$FeSO_4 \rightarrow Fe_2O_3 + SO_2 + SO_3 \quad (3)$$

Table 2. Results of SEM-EDS Analysis of Natural Zeolite-Fe

Element	Percentage (%)
O	53.09
Si	22.78
Al	6.22
Fe	6.2
Na	2.08
K	1.74
Ca	3.6
S	4.29

Table 2 presents the EDS results of natural zeolite-Fe. The Fe content increased to 6.2%, confirming the successful impregnation of Fe into the natural zeolite structure. However, the presence of the sulfur (S) element in the analysis suggests that not all $FeSO_4$ was converted into Fe_2O_3 during thermal treatment. This presence of residual sulfur is potentially detrimental, as it may reduce the photocatalytic effectiveness by partially blocking active sites or interfering with electron transfer processes.



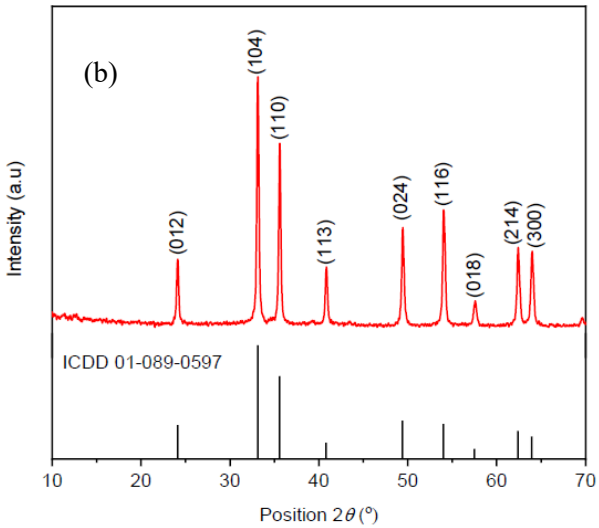


Figure 3. (a) XRD pattern of Natural Zeolite-Fe, (b) XRD pattern of Hematite Nanoparticles with Reference (Ilmi et al., 2021)

The X-ray diffraction pattern (Figure 3. (a)) confirms that the natural zeolite is a mixture of mordenite and clinoptilolite. The presence of mordenite is identified by characteristic diffraction peaks at 2theta = 25.68°, 26.66°, and 27.83°, while clinoptilolite is indicated by peaks at 2theta = 9.08° and 13.27° (Gago & Ngapa, 2021; Ngapa & Ika, 2020). Additionally, the presence of Fe_2O_3 (hematite) is indicated by the appearance of peaks at 35.37° and 64.19° (Ilmi et al., 2021). Most hematite peaks (33°–64°) do not overlap with zeolite peaks, allowing them to be distinguishable.

Fenton Photo Test

Determination of the Optimal Condition

The results of the decolorization of methylene blue under different conditions are presented in Figure 4. The tested conditions include: (i) without H_2O_2 & UV, (ii) with H_2O_2 & without UV, and (iii) with H_2O_2 & UV irradiation. As shown in Figure 4, the highest decolorization percentage of methylene blue is achieved under the combination of H_2O_2 concentration of 30 mM and under 365 nm UV lamp irradiation, reaching 99.77% after a contact time of 120 minutes for an initial methylene blue concentration of 20 mg/L. Under conditions with H_2O_2 but without UV, the

decolorization percentage reaches 90.15%, while without H_2O_2 & UV, the decolorization percentage is 62.85%.

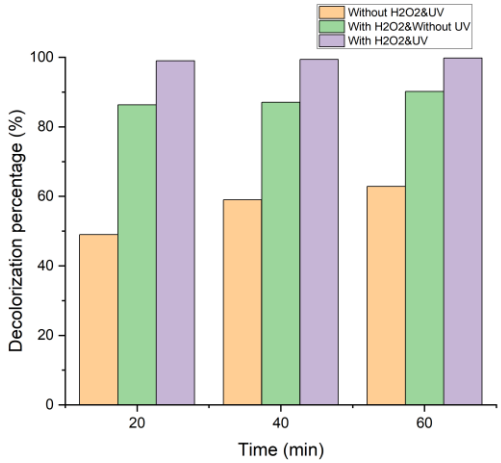


Figure 4. Effect of Different Conditions on the Decolorization Percentage (pH= 3; Dose of Natural Zeolite-Fe= 0.05 grams; H_2O_2 Concentration= 30 mM; Methylene Blue Concentration= 20 mg/L; V= 500 mL)

These results indicate that the decolorization process of methylene blue using natural zeolite-Fe involves both adsorption and photo-Fenton mechanisms. The significant decolorization observed even without H_2O_2 & UV suggests that adsorption plays a role in the removal of methylene blue. However, the increased decolorization percentage in the presence of H_2O_2 & UV confirms the contribution of the photo-Fenton process, wherein hydroxyl radicals degrade the methylene blue adsorbed on the natural zeolite surface. The result is in line with the research conducted by Ikhlaq et al. (2019). The improved effectiveness under its condition can be attributed to the presence of Fe, which serves as active sites for the decomposition of H_2O_2. This reaction generates hydroxyl radicals (•OH) as highly reactive oxidizing species, and UV light accelerates the formation of hydroxyl radicals, thereby enhancing the photo-Fenton reaction percentage.

The Effect of pH

The results of decolorization of methylene blue at different pH levels (3, 5, and 7) are presented in Figure 5. This study was conducted under optimal conditions, namely with H_2O_2 and UV. The goal of varying the pH is to evaluate its influence on the performance of natural zeolite-Fe in the photo-Fenton process.

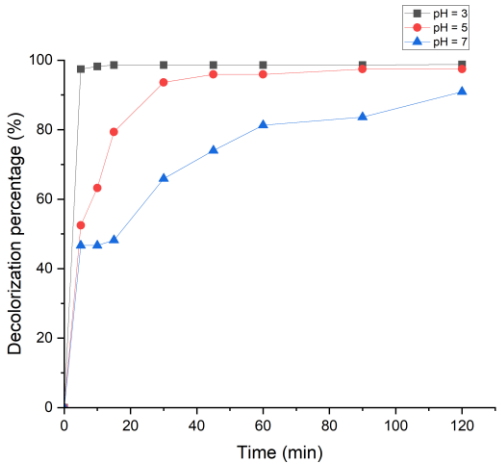


Figure 5. The Effect of pH on the Decolorization Percentage Under Conditions with H_2O_2 & Under 365 nm UV Lamp Irradiation (Natural Zeolite-Fe Dose = 0.05 grams; H_2O_2 concentration = 30 mM; Methylene Blue concentration = 20 mg/L; V = 500 mL)

Figure 5 shows that the highest decolorization percentage is achieved at pH 3, reaching 99.77% after 120 minutes. At pH 5 and pH 7, the decolorization percentages are 97.46% and 90.92%, respectively. These results indicate that the lower the pH, the higher the decolorization percentage.

The optimal condition is at pH 3, in line with the findings conducted by Foko et al. (2025). The decolorization percentage of quinoline yellow at pH 3 is 96.10% after 240 minutes. It means that the formation of hydroxyl radicals is influenced by the pH level.

Acidic condition (around pH 3) promotes the effective decomposition of H_2O_2 by Fe ions, leading to a higher concentration of reactive radicals for dye degradation (Wardiyati et al., 2012). At pH levels higher than 3 but below neutral, the oxidation efficiency decreases due to the reduced formation rate of hydroxyl radicals, primarily resulting from the hydrolysis of Fe^{2+} ions. The hydrolysis reactions of Fe^{2+} are as follows (Gea et al., 2020).

$$Fe^{2+} + H_2O \rightleftharpoons FeOH^+ + H^+ \quad (4)$$

$$Fe^{2+} + 2H_2O \rightleftharpoons Fe(OH)_2 + 2H^+ \quad (5)$$

$$Fe^{2+} + H_2O \rightleftharpoons Fe(OH)^{3-} + 3H^+ \quad (6)$$

At neutral to basic pH, H_2O_2 becomes unstable, rapidly decomposes into water and oxygen, and this

decomposition reduces the availability of H_2O_2. Moreover, the formation of hydroxyl radicals will be hindered due to the formation of $Fe(OH)_3$ (Hayati et al., 2021).

$$H_2O_2 \rightarrow H_2O + O_2 \quad (7)$$

$$Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3 \quad (8)$$

The Effect of H_2O_2 Concentration

The results of decolorization of methylene blue at different H_2O_2 concentrations (15, 30, and 45 mM) are presented in Figure 6. This study is conducted under optimal conditions, namely with H_2O_2 & UV and pH of 3.

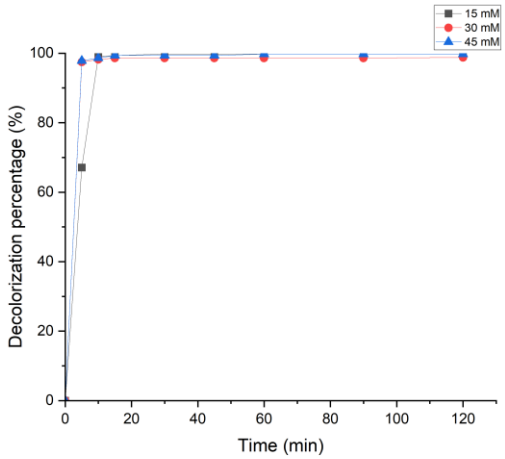


Figure 6. The Effect of H_2O_2 Concentration on the Decolorization Percentage Under Conditions with H_2O_2 & Under 365 nm UV Lamp Irradiation (Natural Zeolite-Fe Dose = 0.05 grams; pH = 3; Methylene Blue Concentration = 20 mg/L; V = 500 mL)

At a contact time of 5 minutes, increasing the H_2O_2 concentration from 15 mM to 45 mM led to an increasing decolorization percentage, from 67.08% to 97.85%. This trend indicates that as the concentration of H_2O_2 increases, the generation of hydroxyl radicals is enhanced, thereby accelerating the decolorization process. This observation is corroborated by the kinetic modeling; the pseudo-second-order rate constant (k_2) increased as the concentration of H_2O_2 increased (Table 3). However, with increasing contact time up to 120 minutes, there is no significant difference in decolorization percentage. The plateau effect occurs because almost all dyes had been decolorized at 120 minutes.

Table 3. Rate Constant Based on Pseudo-Second Order Modelling

Concentration of H_2O_2 (mM)	Rate constant, g $mg^{-1}.min^{-1}$	R^2
15	0.0055	0.9993
30	0.0192	1
45	0.0417	0.9999

COD Analysis

COD analysis is conducted on two samples: (i) the sample before the photo-Fenton process and (ii) the sample after the photo-Fenton process that resulted in the highest decolorization percentage.

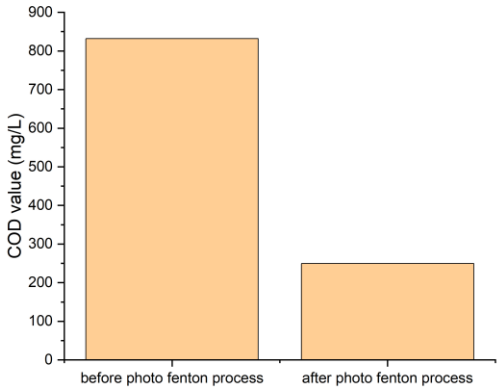


Figure 7. Comparison of COD Value Before and After Photo-Fenton Process

As shown in Figure 7, the COD value only decreased by about 70% from 832 mg/L to 243.6 mg/L, following the photo-Fenton process. It does not meet the threshold value set by PERMENLHK No.5 of 2014, which has a maximum allowable COD value of 150 mg/L. This result contrasts with the high decolorization percentage of 99.77%. In accordance with the study conducted by Peñafiel et al. (2025), the COD value only decreased by about 81.8% from 1,373 mg/L to 230 mg/L after the photocatalysis process, while the decolorization percentage achieved up to 98.8%. This discrepancy suggests two main possibilities. First, the chromophore groups responsible for color are readily broken down by hydroxyl radicals, but the complete mineralization of

organic compounds into CO_2 and H_2O is not fully achieved. To validate this hypothesis, Total Organic Carbon (TOC) analysis is required to quantify the actual extent of mineralization. Second, the residual H_2O_2 interfered with the COD measurement.

CONCLUSION

The iron content of natural zeolite-Fe was 6.2 wt%. The optimal condition for decolorization of methylene blue was under conditions with H_2O_2 and a UV lamp under 365 nm, resulting in a decolorization percentage of 99.77% at 120 minutes. The optimal pH level for decolorization of methylene blue was 3. The decolorization was rapid with increasing H_2O_2 concentrations. This was reflected in the pseudo-second-order rate constant, which increased as the H_2O_2 concentrations increased. Future research is essential to address key limitations, such as investigating the iron leaching, conducting recyclability tests, and quenching H_2O_2 before COD analysis.

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