

The investigation of different decolorization mechanism of methylene blue in the system with and without photocatalyst made from Indonesia laterite ore

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Abstract

Purpose. This work aims to investigate the performance of photocatalyst made from laterite ore to decolorize organic waste of methylene blue.

Methods. Laterite ore was processed through leaching in chloride solution, followed by filtration and neutralization using sodium hydroxide to obtain a precipitate that contains maghemite photocatalyst. The maghemite was characterized to ensure its capability as a photocatalyst. The decolorization experiment using methylene blue as a representation of organic waste was conducted in the absence and presence of a photocatalyst in a constant UV light to reveal the mechanism of methylene blue decolorization. Hydrogen peroxide and pH are controlled to optimize the photocatalytic efficiency.

Findings. A photocatalyst made from laterite contributes to the significant increase in the decolorization degree of methylene blue through the release of hydroxyl radicals, a powerful substance for decomposing organic matter. The optimal decomposition of methylene blue is achieved by combining the increase of hydrogen peroxide and the reduction of the pH of the organic waste. The optimum pH for the decolorization of methylene blue through photocatalytic reaction is at pH = 3.

Originality. For the first time, the mechanism of decolorization in the absence and presence of photocatalyst is revealed, resulting an optimum condition to achieved highest degree of methylene blue decolorization.

Practical implications. The method for synthesizing photocatalysts can be applied to produce alternative products from laterite ore, especially low-grade laterite ore, a by-product of high-grade nickel ore mining. The optimum conditions revealed in this research can be applied to synthesize photocatalysts from laterite ore and to remediate organic wastes.

Keywords: laterite, maghemite, photocatalyst, decolorization

1. Introduction

Dyes, the coloring agents widely used in various chemical industries, are toxic, carcinogenic, non-biodegradable, and high chemical oxygen demand substances [1], [2]. The resistance of dye molecules to light, temperature, and oxidizing agents makes them accumulate in living organisms, causing severe diseases and disorders. Thus, the disposal of wastewater containing dyes from industries harms the environment and humans severely. Various techniques, such as filtration, adsorption, and other chemical and biological treatments, have been used to treat water-containing dyes. Unfortunately, those processes cannot neutralize all organic pollutants [3], [4]. Another method worth applying in the treatment of water-containing dyes is a treatment involving a photocatalysis reaction. Photocatalysis is an advanced oxidation process (AOP) that relies on the in-situ hydroxyl radical production as a substance that neutralizes waste molecules. This method

utilizes non-toxic and chemically stable materials, which are efficient and effective in degrading organic pollutants [5], [6].

Photocatalyst materials were made from various sources, and most of them are materials from chemical manufacturers. The photocatalyst made from chemicals becomes unnatural waste after use, whereas the photocatalyst made from natural resources will coexist with nature after use. Thus, natural material-based photocatalyst is preferred for photocatalyst synthesis. Minerals are among the natural materials that can be used in photocatalyst synthesis. Minerals are vastly available, unique in morphology, mechanically stable, environmentally friendly, and cost-effective natural materials [7]-[9].

Laterite is one of the minerals that has a potential application to be used in photocatalyst synthesis. Laterite ore is the product of intense weathering of silicate rocks influenced by climate, groundwater pH, and geomorphology [10]. The common minerals in laterite are goethite (FeOOH), quartz (SiO₂), and magnesium silicate [11]-[13]. Goethite in laterite

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is the matrix mineral associated with other minerals, implying that laterite ore contains a large amount of iron oxide, a material that can be used to synthesize photocatalyst [14].

Massive laterite deposits can be found in tropical regions such as Indonesia, India, Sri Lanka, Australia, and some countries in the African continent. Many studies have been conducted on encompassing laterite ore potential as an alternative low-cost raw material for industry [15]-[17]. Laterite ore was used to adsorb coloring agent molecules from water-containing dyes without decomposing the coloring agent molecules [18], [19]. The decomposition of the coloring agents can be conducted using a photocatalyst material. Due to its high iron oxide content, the laterite ore can be used as raw material to synthesize a photocatalyst. The synthesis of photocatalyst material from laterite is a rare process, and the studies to observe the effect of process variables on the photocatalytic reaction using a material made from laterite ore are very minimal.

Therefore, our research aims to synthesize a photocatalyst material from laterite ore and use this laterite-made photocatalyst to decompose coloring agent molecules in dyes, emphasizing the effect of process variables in the photocatalytic reaction. Laterite from Indonesia was prepared using simple mineral processing technology to produce a photocatalyst material. The laterite ore was crushed and milled to produce a laterite powder, which was leached, precipitated, and heated before being used as a photocatalyst. The laterite was characterized using various analytical techniques. The photocatalytic reaction using a photocatalyst made from laterite was conducted to neutralize methylene blue ($C_{12}N_2(CH_3)_4NSCl$), a substance often used to represent organic liquid waste. The observed process variables are pH and hydrogen peroxide, which are critical for the photocatalytic reaction.

The reactions of photocatalyst synthesis from laterite are rarely studied, as well as the reaction mechanism of methylene blue decolorization. Hence, the aim of this work is to study the reaction mechanism of photocatalyst synthesis and the photocatalytic reaction of methylene blue decolorization. The effect of nitrogen doping, as well as pH and hydrogen peroxide on the decolorization degree, is also intensively discussed.

2. Methods

2.1. Preparation of laterite based photocatalyst

Dry laterite, obtained from South East Sulawesi, Indonesia, was ground and milled using a laboratory jaw crusher and ball mill, resulting in a powder of laterite ore. Hydrogen chloric acid (HCl, Merck) 16% was used in the leaching stage to dissolve iron from laterite ore. A liter HCl solution was used to dissolve 500 g of laterite ore. The suspension of laterite-HCl solution was stirred for 2 hours at 60-80°C prior to filtering and separation. The sodium hydroxide (NaOH, Merck) was added into the filtrate to obtain iron oxide precipitate. The precipitate was rinsed, dried at 150°C for 19 hours, and calcined at 500°C for 2 hours. The calcined powder was ground to 100 mesh size before being utilized as a photocatalyst.

2.2. Characterization of the as-received laterite ore and the photocatalyst material

Identification of minerals or compounds in the as-received laterite and photocatalyst materials was conducted using X-ray diffraction (XRD, Bruker D8 Advance). A Scanning Electron Microscope (SEM, JEOL JSM IT300) revealed laterite ore and iron oxide photocatalyst morphology. The bonds in molecules of laterite and photocatalyst were detected using Fourier Transfer Infrared (FTIR, Thermo-Scientific Nicolet IS5).

2.3. Photocatalyst performance test

An 8-watt UV lamp was used as the ultraviolet source to conduct a photocatalytic reaction to neutralize methylene blue. The decolorization of methylene blue, which indicates methylene blue degradation, was used as the photocatalyst performance parameter of the photocatalyst made from laterite. The photocatalyst materials at 0.05 to 0.5 mg were poured into 100 mL of 20 ppm methylene blue solution. Before irradiation, the mixture was placed in darkness for 30 minutes to ensure the equilibrium of dye adsorption-desorption on the photocatalyst surface. As the UV lamp was turned on, 0.1, 1.0 and 4.0 mL hydrogen peroxide (H_2O_2 , Merck) were added to the methylene blue solution. The pH of each of these solutions was adjusted to pH of 3, 5, 7, and 10 by adding either NaOH or HCl. At a given time intervals, 2 mL of liquid was picked from the solution for methylene blue concentration measurement. The liquid phase in the samples was separated from solid particles using centrifuge equipment. The concentration of methylene blue in the solution was measured using UV-vis spectroscopy at wavenumber 663 nm. The actual concentration of methylene blue was calculated by comparing the decolorization of standard samples and that of experimental samples. The following Equation was used to calculate the decolorization of methylene blue:

$$\% \text{ decolorization} = \frac{C_i - C_t}{C_i} \cdot 100\%, \quad (1)$$

where:

C_i – initial concentration of methylene blue;

C_t – concentration of methylene blue at a certain time.

3. Results and discussion

3.1. Characterization of Indonesia laterite ore and the photocatalyst made from laterite

The elemental composition of Indonesia laterite ore and photocatalyst made from laterite is shown in Table 1, whereas the minerals or compounds that compose laterite ore are shown in Figure 1. The table shows that laterite ore consists of oxygen, magnesium, and silicon as dominant elements.

Table 1. Elemental composition of laterite ore from Indonesia and photocatalyst made from laterite ore

| Element | O | Fe | Mg | Si | Ni | Al | Cr | Na | Cl |
|------------------------------------------------------------------|------|------|------|------|------|------|------|------|------|
| Weight of element in the as-received laterite ore, % | 51.6 | 19.5 | 12.2 | 12.3 | 2.31 | 1.32 | 0.39 | – | – |
| Weight of element in photocatalyst prepared from laterite ore, % | 42.4 | 21.4 | 18.2 | – | 7.63 | 1.50 | 5.33 | 2.39 | 5.33 |

The XRD pattern of laterite ore presented in Figure 1 shows that laterite ore consists of antigorite ($Mg_3Si_2O_5(OH)_4$), quartz (SiO_2), and goethite ($FeOOH$). Our analysis of laterite ore is consistent with previous research showing that goethite, quartz, and magnesium silicate are the common minerals in laterite ore [11]-[13]. The previous research showed that goethite can be used to adsorb various organic substances, and it can also be used as a photocatalyst due to its low bandgap, which is 2.1 eV [20]-[22].

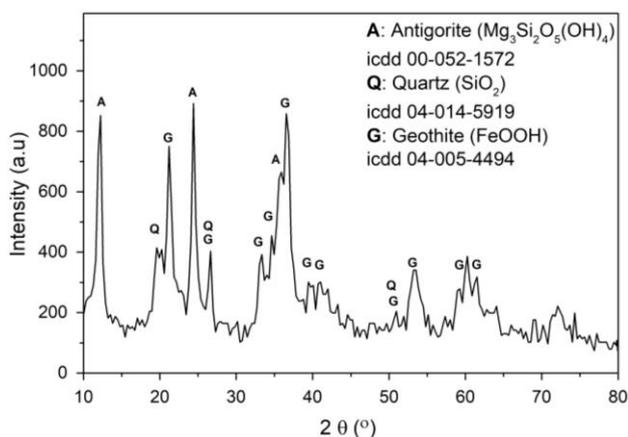


Figure 1. XRD pattern of Indonesia laterite ore

However, our preliminary experiment involving goethite in laterite to decolorize methylene blue as a waste organic model shows that adsorption of methylene blue occurred without significant photocatalytic reaction. Laterite contains a large amount of silicate clay minerals that can adsorb organics. Unfortunately, after being adsorbed, the adsorbed methylene blue on the surface of silicate clay sticks to the goethite surface, resulting in a slowdown in the photocatalytic reaction of methylene blue decolorization. Therefore, goethite must be separated from silicate clay to make it able to conduct a continuous photocatalytic reaction.

The removal of silicate clay minerals results in goethite as a photocatalyst material. The elemental composition and XRD pattern of this photocatalysts are presented in Table 1 and Figure 2, respectively. Silicate clay minerals and silicon oxide were removed from the as-received laterite, increasing other metallic elements in the photocatalyst material.

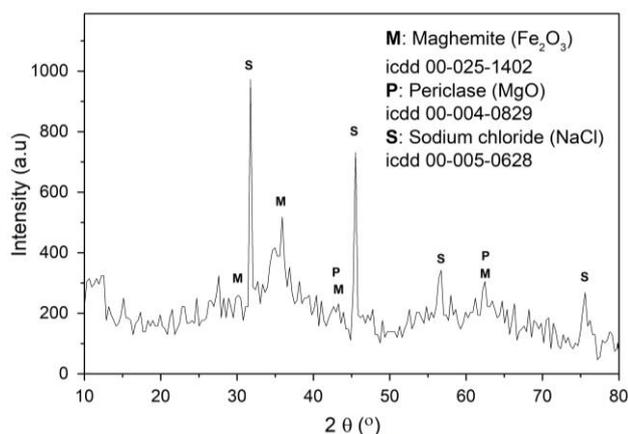
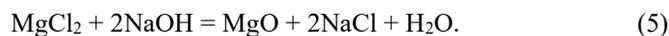
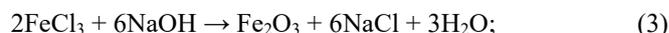


Figure 2. XRD pattern of Photocatalyst made from Indonesia laterite ore

The XRD patterns in Figure 2 show that maghemite (γ-Fe₂O₃), periclase (MgO), and sodium chloride (NaCl) were formed after processing laterite ore to become photocatalysts. The type of minerals in precipitate depends on the type of solvent in the leach solution and the reagent to neutralize the solution. For example, the precipitate of sulphate leach solution neutralized by sodium hydroxide contains ferrihydrite (Fe³⁺)₂O₃·0.5H₂O, goethite (FeOOH), schwertmannite (Fe₈O₈(OH)₆(SO₄)·nH₂O), and sodium sulphate (Na₂SO₄) [23]. On the other hand, the precipitate of chloride leach solution, neutralized by hydrogen chloride, contains

hematite or maghemite (Fe₂O₃), periclase (MgO), and sodium chloride [24], [25]. Hence, the composition of minerals in precipitate synthesized from chloride leach solution is consistent with previous research.

The following Equations are the possible chemical reactions during the synthesis of photocatalysts:



Maghemite consists of iron oxide with a chemical composition that resembles hematite (γ-Fe₂O₃) but possesses a molecular structure like magnetite (Fe₃O₄). Unlike magnetite, which contains trivalent and divalent iron ions, maghemite contains only trivalent ions [26]. The bandgap of maghemite is relatively low, 2.47 eV, which makes it applicable as a photocatalyst material [27].

Figures 3 and 4 present the photocatalyst's morphology and particle size distribution, respectively. Morphology and particle size distribution of powder are the essential factors in heterogeneous reactions, especially in a catalytic reaction. The surface of photocatalyst particles exhibits high porosity, a rough texture, and various irregular particle shapes. Some particle aggregation is observed, likely due to the welding effect among particles during milling. The particle size analysis of the photocatalyst powder shows that the particle size is in the range of 0.25 to 500 μm with an average size of 28 μm. Since the standard average size of catalyst particles is 3.5-30 μm [28], the photocatalyst particles prepared from Indonesia laterite ore have the recommended size for a catalyst.

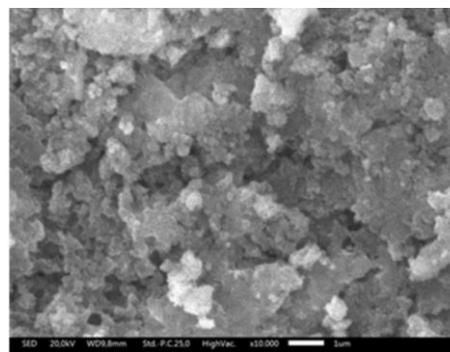


Figure 3. The morphology of photocatalyst prepared from Indonesia laterite

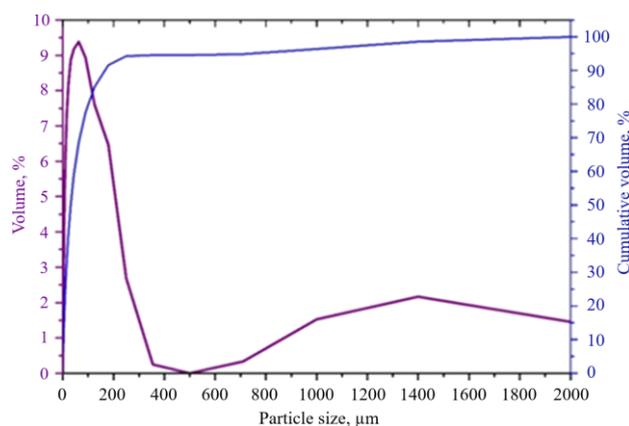


Figure 4. The Particle size distribution of photocatalyst prepared from Indonesia laterite

Figure 5 displays the FTIR spectra of Indonesia laterite ore and photocatalyst within the 4000-400 cm^{-1} and that of photocatalyst at low wavenumber range. The stretching vibration peak at 3683 cm^{-1} denotes serpentine antigorite in nickel laterite, linked to hydroxyl groups with magnesium [29], [30]. In contrast, the absence of the 3683 cm^{-1} band in the photocatalyst indicates the decomposition of $\text{Mg}(\text{OH})_2$ to MgO . The formation of MgO facilitates the adsorption of H_2O and CO_2 , resulting in a broad band at 3400 cm^{-1} associated with OH stretching vibrations of adsorbed water molecules. Additionally, a weaker band at 2360 cm^{-1} reflects the adsorption of gas-phase CO_2 . A bending vibration of O-H bonds is evident at 1629 cm^{-1} , likely resulting from surface hydration [31]-[33]. The peak at 992 cm^{-1} in laterite ore corresponds to Si-O-Si bond vibrations, while the peak at 790 cm^{-1} relates to the elongation of AlO, SiO, or MgO bonds [30].

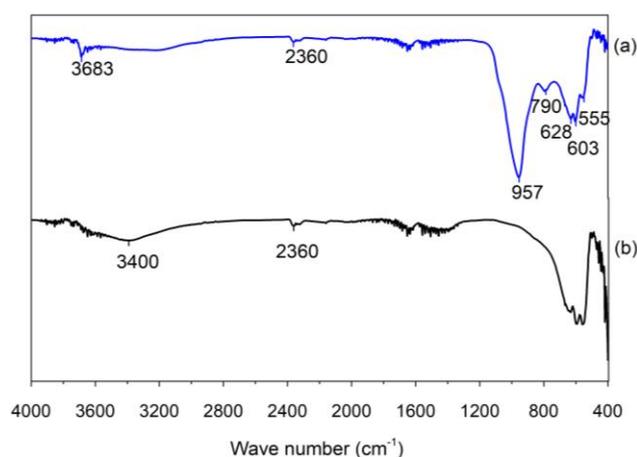


Figure 5. FTIR spectra of Indonesia laterite ore (a) and photocatalyst at wide range wavenumber (b)

In Figure 6, The FTIR analysis of photocatalyst in the low wavenumber region (800 to 400 cm^{-1}) reveals several distinct peaks in the low wavenumber region (800 to 400 cm^{-1}), attributed to characteristic $\gamma\text{-Fe-O}$ vibration bands [34], [35]. Furthermore, a band around 630 cm^{-1} signifies the maghemite phase of iron oxide ($\gamma\text{-Fe}_2\text{O}_3$) [34], [36].

3.2. Effect of photocatalyst material quantity, hydrogen peroxide concentrations and pH on methylene blue decolorization

The effect of photocatalyst quantity, hydrogen peroxide concentrations, and initial pH on methylene blue decolorization is presented below. Figure 7 shows that an increase of photocatalyst quantity up to 1.0 g increased the % decolorization. This improvement in decolorization efficiency suggests that higher photocatalyst quantity enhances the production of hydroxyl radicals and provides more active sites for adsorption. However, an increase in photocatalyst quantity of more than 1.0 g resulted in a decrease of % decolorization. The decrease in % decolorization at the high quantity of photocatalyst is possibly caused by the agglomeration of particles and the ultraviolet light retention at the high population of particles.

The figure also shows that the highest % decolorization of methylene blue is 17.81% for 6 hours, which is a low value of methylene blue decolorization. Hence, an effort is needed to improve the photocatalytic reaction of methylene blue decolorization.

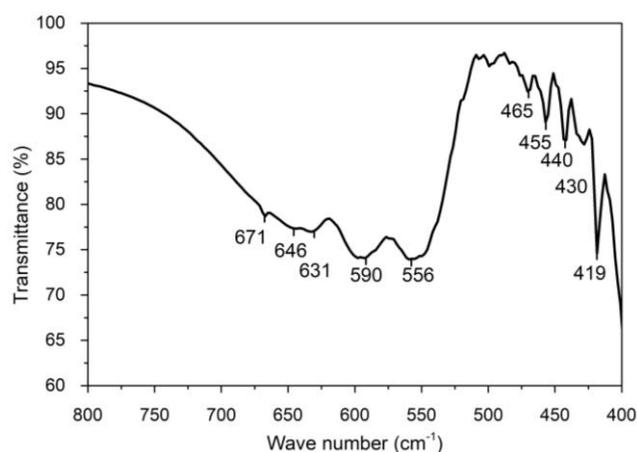


Figure 6. FTIR spectra of photocatalyst at low wavenumber range

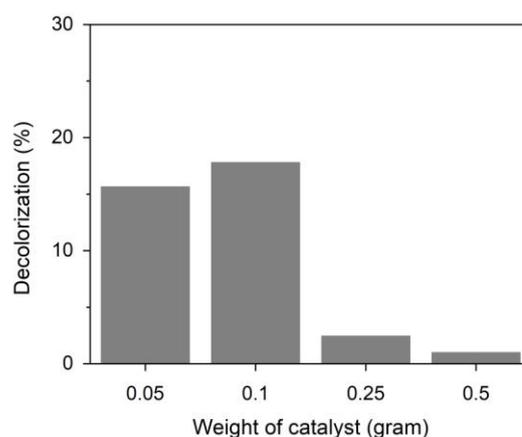
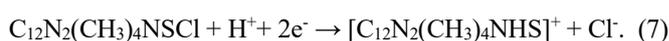


Figure 7. The effect of quantity of photocatalyst on the % decolorization of methylene blue

The addition of hydrogen peroxide can increase the % decolorization of methylene blue. Hydroxyl radical is a solid oxidizing species with a reduction potential of 2.8 V, which can oxidize dyes into H_2O , CO_2 , and mineral acids [37], [38]. Adding hydrogen peroxide (H_2O_2) can improve photocatalytic reaction by rapidly forming hydroxyl radical ($\bullet\text{OH}$) and superoxide ions, the species that can react and degrade methylene blue, increasing % decolorization of methylene blue.

Figure 8 shows that the addition and the increase of hydrogen peroxide can increase the % decolorization of methylene blue. The figure also shows that the decolorization of methylene blue occurred in both systems with and without a photocatalyst. Previous research indicated that hydrogen peroxide decomposes organic compounds with a photocatalyst [39]-[41]. The methylene blue decolorization with the presence of photocatalyst was contributed by the formation of hydroxyl radical as the product of hydrogen peroxide reduction by the electrons from the conduction band of photocatalyst atoms [42], [43].

On the other hand, the absence of photocatalyst materials makes it challenging to produce hydroxyl radicals; hence, the decolorization of methylene blue in the system without a photocatalyst must occur by a different mechanism. The possible mechanism for decolorizing methylene blue without a photocatalyst is that hydrogen peroxide acts as the reducing agent for methylene blue through the following reaction Equations:



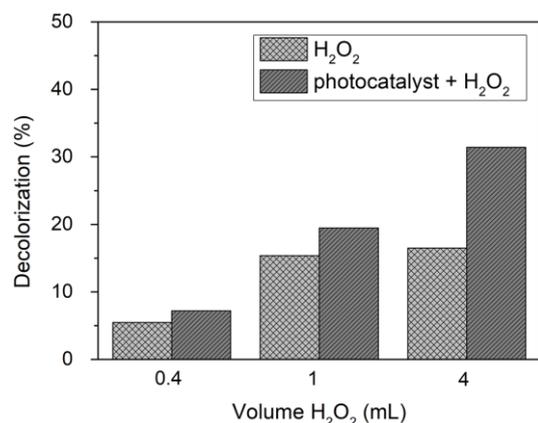


Figure 8. The effect of volume of hydrogen peroxide on the % decolorization of methylene blue

Increased hydrogen peroxide in the methylene blue solution should lead to rapid decolorization. However, Figure 8 shows that without a photocatalyst, the decolorization degree of methylene blue reached a slightly different value of 15.37% after adding 1 mL and 16.51% after adding 4 mL hydrogen peroxide during 3 hours. Decolorization without a catalyst degraded methylene blue by 16.51% and increased to 31.41% in the presence of a photocatalyst. The stability of hydrogen peroxide from oxidation reaction at low pH increased as the pH in solution decreased [44], [45]. The reaction in Equation (7) shows that hydrogen ions, as the product of the reaction in Equation (6), are involved in decolorization of methylene blue and the formation of hydrogen chloride (HCl). The formation of hydrogen chloride eventually decreased the pH of the methylene blue solution, stabilizing hydrogen peroxide and causing a reaction in Equation (6) to decline and, in turn, to stop the decolorization reaction of methylene blue.

The presence of photocatalyst materials in methylene blue solution allows the formation reaction of hydroxyl radical ions that decolorize methylene blue. In contrast with the decolorization mechanism without a photocatalyst that stopped when the pH of the solution was too low, the decolorization using a catalyst becomes rapid at low pH. Based on the general photocatalytic mechanism proposed elsewhere, the decolorization of methylene blue in the presence of an iron oxide photocatalyst can be written as follows [42].



Reaction in Equation (9) shows that electrons from the conduction band of photocatalyst atoms and hydrogen ions reacted with dissolved oxygens to form hydroperoxyl radical (HO_2^\bullet) [46]. Hydroperoxyl radical is a crucial intermediate phase in hydroxyl radical formation that can decolorize methylene blue. The formation of hydroperoxyl needs the presence of significant amount of free hydrogen ions. At neutral or near neutral pH, where free hydrogen ions are rare or limited, the reaction in Equation (7) resulted in the decolorization of methylene blue at only 31.41-32.53% for 3 hours, shown in Figure 9.

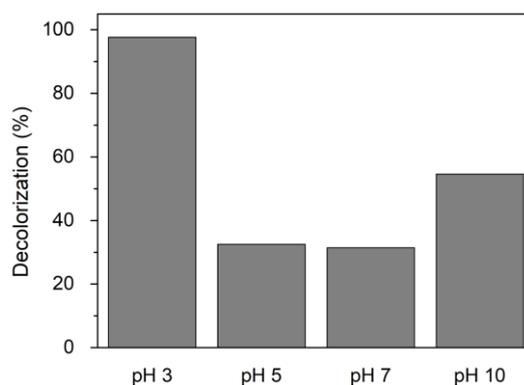


Figure 9. The effect of pH on the % decolorization of methylene blue

The low value of decolorization was caused by the limitation of hydrogen ions. In contrast, at very low pH, where hydrogen ions are available in large amounts, the decolorization of methylene blue achieved 97.67%. Besides contributed by the rapid formation of hydroperoxyl radical at low pH, which in turn becomes hydroxyl radical, the high decolorization at low pH is also contributed by the reaction between hydrogen peroxide and the electrons from the conduction band of photocatalyst atoms that produce hydroxyl radicals [42], [45].



Figure 9 shows that methylene blue decolorization at high pH is 54.57%. Since the reaction in Equation (9) needs a significant amount of hydrogen ions, the reaction was unlikely to continue at high pH. Hence, the rapidity of methylene blue decolorization at high pH is contributed by the role of hydrogen peroxide as a reducing agent (reactions in Equation (6) and (7)) and the reduction of hydrogen peroxide to produce hydroxyl radicals (reaction in Equation (12)).

Methylene blue is among the organic pollutants generated by industries. Other organic pollutants also harm the environment. Among them are methylene orange, benzenes, phenols, waste oil, and dyes. The photocatalyst has potential applications to remediate these pollutants. However, since each type of waste has different conditions, further field experiment is needed before applying this photocatalyst to handle these wastes. Some waste needs pretreatment before being treated by a photocatalyst. The remediation plant also needs to be designed based on the waste condition and the optimization condition of the photocatalyst.

4. Conclusions

Laterite ore consisting of goethite (FeOOH), quartz (SiO_2), and silicate minerals can be processed to make a photocatalyst material of maghemite ($\gamma\text{-Fe}_2\text{O}_3$) powder with an average size of 28 μm and a wavenumber band at around 630 cm^{-1} . The quantity of photocatalyst has the contrast effect, as an increase of photocatalyst quantity increases areas for photocatalytic reaction, resulting in the rapid decolorization; at the same time, the supersaturated photocatalyst powder reduces the flux of ultraviolet light needed for photocatalytic reaction. The decolorization of methylene blue with hydrogen peroxide alone depends on the role of hydrogen peroxide as a reducing agent that works well at neutral or high pH.

The maximum % decolorization using only hydrogen peroxide is 16.51%. On the other hand, the decolorization of methylene blue using hydrogen peroxide and photocatalyst

material depends on the formation of hydroxyl radicals that can decompose methylene blue. In the system with a photocatalyst, at low pH, hydrogen peroxide undergoes a reduction reaction with electrons from the conduction band of photocatalyst atoms to produce hydroxyl radicals, whereas, at high pH, hydroxyl radicals play the role of a reducing agent to decompose methylene blue. Using the combination of a photocatalyst and hydrogen peroxide, the decolorization of methylene blue at low and high pH is 97.67 and 54.57%, respectively.

Author contributions

Conceptualization: AL, HIW, SS; Data curation: AL, HIW, END; Formal analysis: AL, END; Funding acquisition: SS; Investigation: AL, END; Methodology: AL, END, SS; Project administration: SS; Resources: SS; Software: AL, SS; Supervision: SS; Validation: AL, END, MR, MDY, SS; Visualization: AL, SS; Writing – original draft: AL, SS; Writing – review & editing: AL, MR, MDY, SS. All authors have read and agreed to the published version of the manuscript.

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Conflicts of interests

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

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Дослідження різних механізмів знебарвлення метиленового синього в системі з фотокаталізатором з латеритної руди Індонезії та без нього

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Мета. Метою роботи є дослідження ефективності фотокаталітичного матеріалу з латеритної руди та його використання для розкладання молекул барвників з особливим акцентом на вплив змінних процесу в фотокаталітичній реакції.

Методика. Латеритну руду переробляли шляхом вилуговування у хлоридному розчині з подальшою фільтрацією та нейтралізацією із використанням гідроксиду натрію для отримання осаду, який містить фотокаталізатор магеміт. Магеміт був охарактеризований для забезпечення його здатності як фотокаталізатора. Експеримент зі знебарвлення із використанням метиленового синього як представника органічних відходів проводили за відсутності та наявності фотокаталізатора в постійному ультрафіолетовому світлі, щоб виявити механізм знебарвлення метиленового синього. Перекис водню і рН контролювалися для оптимізації фотокаталітичної ефективності.

Результати. Встановлено, що фотокаталізатор, виготовлений з латериту, сприяє значному підвищенню ступеня знебарвлення метиленового синього завдяки вивільненню гідроксильних радикалів – потужної речовини для розкладання органічних речовин. Виявлено, що оптимальне розкладання метиленового синього досягається шляхом поєднання збільшення кількості перекису водню та зниження рН органічних відходів. Визначено оптимальний рівень рН для знебарвлення метиленового синього шляхом фотокаталітичної реакції, який становить рН = 3.

Наукова новизна. Вперше виявлено механізм знебарвлення за відсутності та наявності фотокаталізатора, внаслідок чого досягається оптимальна умова для досягнення найвищого ступеня знебарвлення метиленового синього.

Практична значимість. Метод синтезу фотокаталізаторів може бути застосований для виробництва альтернативних продуктів з латеритної руди, особливо низькосортної, яка є побічним продуктом видобутку високоякісної нікелевої руди. Оптимальні умови, виявлені в цьому дослідженні, можуть бути застосовані для синтезу фотокаталізаторів з латеритної руди та для ремедіації органічних відходів.

Ключові слова: латерит, магеміт, фотокаталізатор, знебарвлення

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