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Effectiveness of Montmorillonite-ZnO as Photocatalyst for Adsorption and Degradation of Methylene Blue

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Abstract

This study aims to synthesize, characterize, and evaluate the photocatalytic activity of a montmorillonite–ZnO (MMT/ZnO) composite for the degradation of methylene blue (MB) under ultraviolet (UV) and dark (non-UV) conditions. The composite was prepared by mixing synthetic montmorillonite derived from Indonesian soil with ZnO in ethanol, followed by calcination at 400 °C for 2 hours, and subsequently characterized using X-ray fluorescence (XRF). The degradation tests were performed using various composite masses (0.5–2.5 g) and compared with single ZnO and montmorillonite materials through visible spectrophotometry analysis. The results revealed that under UV irradiation, MMT/ZnO achieved the highest degradation efficiency of 98.37%, while under non-UV conditions, MB removal remained high at 88.39%, primarily driven by adsorption. These findings confirm a synergistic effect between the adsorption capacity of montmorillonite and the photocatalytic activity of ZnO, where adsorption dominated MB removal while photocatalysis contributed to enhancing overall degradation efficiency. The novelty of this study lies in the utilization of synthetic Indonesian montmorillonite as a ZnO support, which enhances photocatalytic efficiency while reducing production costs. This finding highlights the potential of MMT/ZnO composites as an environmentally friendly and cost-effective alternative for dye wastewater treatment in the textile industry.

Keywords: Montmorillonite, ZnO, Photocatalysis, Methylene Blue, Adsorption, Degradation

INTRODUCTION

Methylene blue is one of the most widely used synthetic dyes in the textile industry (Karuppasamy et al., 2021). In the dyeing process, only about 5% of the methylene blue dye is utilized, while the remaining 95% is discharged as waste (Kustiningsih et al., 2017). Referring to the Decree of the Minister of Environment concerning the quality standards for industrial wastewater (KEP-51/MENLH/10/1995), the permissible concentration of methylene blue in water ranges from 5 to 10 mg/L. This dye possesses toxic, carcinogenic, mutagenic, and highly persistent characteristics, making it likely to remain in the environment for a long time (Ćurković & Dobrović, 2010). Due to its stability, methylene blue is not easily degraded naturally. This leads to the accumulation of harmful substances in water channels and soil. Pollution caused by wastewater containing methylene blue not only reduces water quality but can also disrupt aquatic ecosystems and pose potential health risks to humans and animals (Khan et al., 2022). Several wastewater treatment techniques have been applied to address pollutants from the textile industry, including adsorption, chlorination, ozonation, and biodegradation. These methods require relatively high operational costs, making them less effective for application in Indonesia (Fatimah et al., 2006). One of the relatively inexpensive and easily applicable methods is photodegradation, which utilizes photocatalysts derived from semiconductor materials such as TiO_2, ZnO, Fe_2O_3, CdS, and others (Sakthivel et al., 2003).

The effectiveness of semiconductor photocatalysts in decomposing organic pollutants has been extensively studied. Although TiO_2 is known as an excellent photocatalyst, its low quantum efficiency limits its practical applications (Li et al., 2022). As an alternative, ZnO offers advantages such as high physical and chemical stability, diverse morphology, non-toxic properties, and low production cost (Di Mari et al., 2022). However, due to its wide band gap (~3.03–3.37 eV), ZnO exhibits limited light absorption in the visible region (Chu et al., 2014; Hasim et al., 2014). In addition, the high recombination rate of electron–hole pairs generated by light in single ZnO limits its large-scale application (J. Jiang et al., 2017). Therefore, surface modification of ZnO is essential to enhance its photodegradation activity. One effective method is modification with montmorillonite (MMT). MMT consists of one octahedral layer of Al^{3+}

sandwiched between two tetrahedral layers of Si^{4+}. MMT can bind various inorganic cations within its layers and adsorp negatively charged species (D. Bin Jiang et al., 2019). According to Julita et al. (2022), the modification of ZnO with Au particles reduces the band gap energy from 3.23 eV to 3.17 eV, strengthens light absorption, and decreases the recombination of electron–hole pairs, thereby enhancing photocatalytic performance. Based on these findings, MMT can be utilized as a promising material for the removal of cationic dyes such as methylene blue from textile industry wastewater.

Compared with other materials such as zeolite or biochar, montmorillonite (MMT) was selected because it possesses a flexible layered structure, a large specific surface area, and a high cation-exchange capacity (D. Bin Jiang et al., 2019). This layered structure allows ZnO particles to be more uniformly dispersed within the MMT galleries, preventing agglomeration and enlarging the active photocatalytic area (Pannak et al., 2018). In addition, MMT exhibits better thermal and chemical stability than amorphous biochar and provides more open pores than rigid zeolite (Fatimah et al., 2021). These characteristics make MMT an ideal support for enhancing the photocatalytic efficiency of ZnO in dye degradation processes.

Montmorillonite (MMT) is the main component of bentonite and has a layered and porous structure. In addition, MMT possesses a large surface area, good adsorption capacity and cation-exchange ability, and low cost (Dao et al., 2023). The main advantage of MMT is its ability to adsorb and bind various organic and inorganic substances, so it has been widely applied in various industries such as water treatment, agriculture, pharmaceuticals, cosmetics, and advanced materials. Several studies have combined ZnO with other semiconductors such as MMT, zeolite, and carbon to further enhance its photocatalytic activity (Fatimah et al., 2021; Pannak et al., 2018; Sohrabnezhad & Seifi, 2016). The findings of Tehubijuluw et al. (2023) showed that ZnO/ZSM-5 achieved a photodegradation efficiency of 98.38% at pH 11 with a surface area of 277.55 m^2/g, confirming that the support of porous materials can increase the contact between the catalyst and dye molecules.

The study by Dao et al. (2023) successfully immobilized ZnO on the surface of MMT through a chemical synthesis method. The synthesized product was used as a photocatalyst for the degradation of rhodamine B. Rhodamine B, methyl orange, and methylene blue are among the components found in industrial wastewater. The MMT used in this study was synthetic MMT (Clay + Imun) derived from Indonesian soil. This synthetic MMT contains Ca, Mg, Fe, Zn, Se, and Al, C, Fe, K, Mg, Na, O, and Si. Therefore, this study aimed to synthesize and characterize MMT–ZnO as a photocatalyst for degrading methylene blue. The synthesized MMT–ZnO was then characterized using the SRF method. In addition, the effects of various operational parameters on the photodegradation efficiency of methylene blue, such as pH, MMT/ZnO content, and initial dye concentration, were also investigated using a visible spectrophotometer.

The purpose of this study is to synthesize a montmorillonite–ZnO composite material and to evaluate its photocatalytic activity in degrading methylene blue under UV and non-UV irradiation, as well as to compare its performance with single ZnO and single montmorillonite to determine the contributions of adsorption and photodegradation at various composite masses.

METHODOLOGY

Materials and Instrumentals

This study was designed as a laboratory experimental research using a completely randomized design (CRD). The treatments consisted of variations in the mass of the MMT/ZnO composite (0.05 g, 0.1 g, 0.15 g, 0.2 g, and 0.25 g). Each treatment was replicated three times to ensure the consistency of the observations. The experiment also included a control in the form of a methylene blue solution without the addition of photocatalyst (negative control) to observe natural degradation, as well as single ZnO and single MMT as comparators. This design enabled the evaluation of the role of each component in the adsorption and photodegradation processes. The test results were compared based on the degradation efficiency obtained from each treatment to assess the effectiveness of the MMT/ZnO composite.

The main instruments used included an analytical balance, beakers, Erlenmeyer flasks, stirring rods, a hot plate stirrer, an oven, a furnace, a volumetric pipette, a photodegradation reactor equipped with a UV lamp, a centrifuge, and a visible light spectrophotometer. The materials used consisted of synthetic montmorillonite, zinc oxide (ZnO), ethanol (C_2H_5OH), 0.1 M NaOH solution, methylene blue, and distilled water.

Methods

Synthesis of MMT/ZnO

The ZnO/MMT photocatalyst was synthesized by mixing 2 g of synthetic montmorillonite with 1 g of

ZnO. Subsequently, 15 mL of 96% ethanol was added to the mixture. The synthesis process was continued by heating and stirring the mixture at 50 °C for 2 hours to ensure homogeneous mixing. Afterward, 0.1 M NaOH solution was added to the mixture to facilitate the precipitation process. The precipitate formed was then separated and calcined at 400 °C for 2 hours to enhance the stability and photocatalytic effectiveness of the material produced.

Characterization of the MMT/ZnO Composite

The synthesized ZnO/MMT photocatalyst was characterized to determine the success of the composite formation. The analysis was carried out using X-ray fluorescence (XRF) to determine the chemical composition of the synthesized MMT–ZnO composite.

Optimization Test of Methylene Blue Photodegradation

The photodegradation optimization of the dye was carried out using methylene blue (MB). A total of 50 mL of MB solution with a concentration of 10 ppm was prepared. Subsequently, varying masses of the ZnO/MMT photocatalyst (0.05 g, 0.1 g, 0.15 g, 0.2 g, and 0.25 g) were added to the solution. The mixture was then placed in the photodegradation reactor. The degradation process was conducted under UV irradiation for 150 minutes with continuous stirring to ensure optimal interaction between the photocatalyst and the dye. After irradiation, the solution was separated by centrifugation at 350 rpm for 30 minutes to obtain a clear supernatant. The absorbance of the remaining dye was then measured using a visible spectrophotometer at the maximum wavelength (665 nm) to determine the degradation efficiency. The degradation efficiency was calculated using the following equation:

$$\%Degradation = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

where:
C_0 = initial concentration of methylene blue (mg/L)
C_t = concentration of methylene blue at a given time (mg/L)

Photocatalytic Effectiveness Test of ZnO/MMT

The effectiveness and efficiency tests of the ZnO/MMT photocatalyst were carried out using methylene blue (MB) as a model pollutant. An MB solution with a concentration of 10 ppm was prepared as the test sample. The degradation effectiveness was then compared under different conditions: the use of montmorillonite + ZnO (MMT/ZnO) under UV light, ZnO under UV, montmorillonite under UV, and the use of montmorillonite + ZnO (MMT/ZnO) without UV light, ZnO without UV, and montmorillonite without UV. The analytical results were compared based on the degradation efficiency values obtained from each treatment to determine the superiority of the ZnO/MMT photocatalyst.

RESULTS AND DISCUSSION

Activation of Montmorillonite/ZnO (MMT/ZnO)

The mixing of 2 g of montmorillonite (MMT) with 1 g of ZnO in 15 mL of ethanol produced a suspension when stirred for 2 hours at 50 °C. Stirring at this moderate temperature serves to open the montmorillonite layers so that ZnO particles can be dispersed into the interlayer spaces. The results showed a yellowish-white suspension with no agglomerates. After homogenization, 0.1 M NaOH solution was added to the suspension to induce the formation of a fine white precipitate, which was then separated. The reaction that occurred is as follows:

$$Zn^{2+}(aq) + 2OH^-(aq) \rightarrow Zn(OH)_2(s) \quad (2)$$

The hydroxide ions (OH^-) from NaOH react with zinc ions (Zn^{2+}) to form zinc hydroxide precipitate ($Zn(OH)_2$). This precipitate is later calcined to produce ZnO, which strongly binds to the surface of MMT. In addition, the OH^- ions can also neutralize weak acidic groups such as –Si–OH or –Al–OH that may still be present on the surface of the synthesized montmorillonite.

The calcination process at 400 °C for 2 hours was carried out to remove residual ethanol and water from the $Zn(OH)_2$ precipitate while simultaneously crystallizing ZnO. After calcination, the material turned bright white with a more porous surface, indicating the release of gases (H_2O and CO_2) during decomposition. In this process, the structure of montmorillonite remained preserved, while ZnO exhibited an increase in crystal density.

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Figure 1. Composite product before grinding

Figure 2. Composite product after grinding

The synthesized product was a composite of montmorillonite/ZnO, with a uniform distribution of ZnO particles and a stable MMT structure. This condition is expected to enhance the interaction between the photocatalyst surface and methylene blue molecules, as well as facilitate electron–hole transfer at the MMT–ZnO interface under UV/Vis light exposure.

Characterization of the MMT/ZnO Composite

The characterization of the MMT–ZnO composite using X-ray fluorescence (XRF) aimed to determine the elemental and oxide composition of the synthesized material. Based on the XRF analysis results (Figure 3), the composite was dominated by zinc (Zn) with a concentration of 75.68%, equivalent to 65.07% in the form of zinc oxide (ZnO). This indicates that the incorporation of ZnO into the montmorillonite matrix was successfully and efficiently achieved.

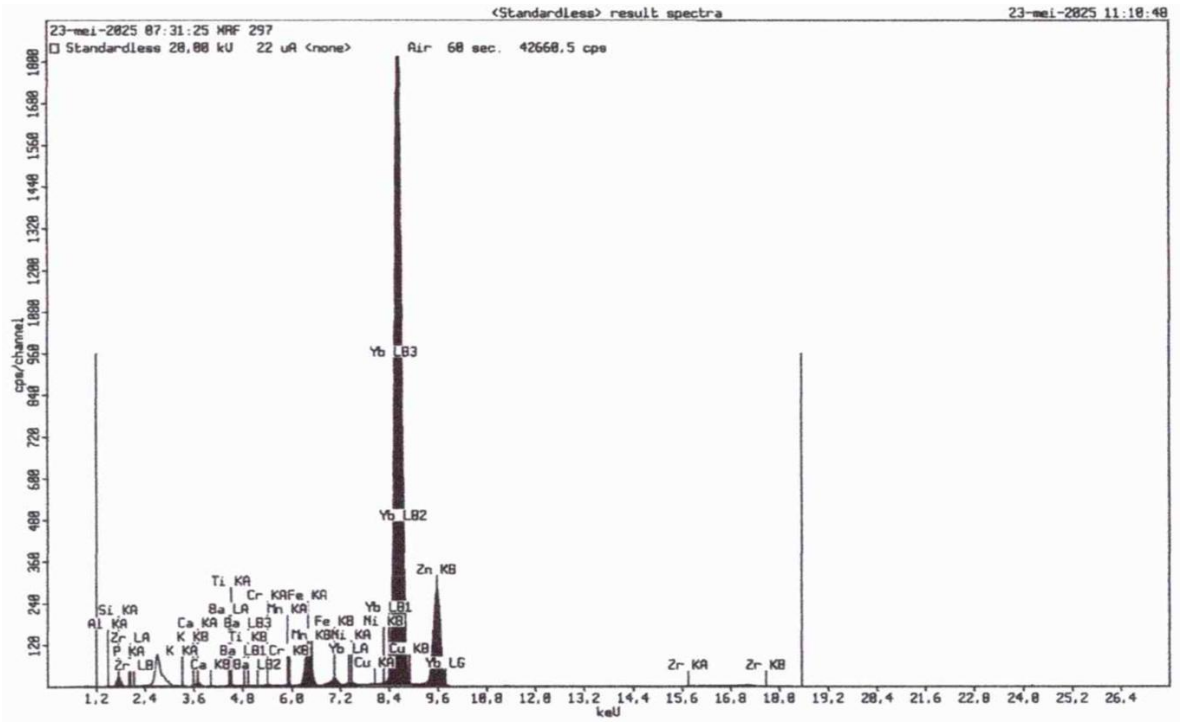

Figure 3. XRF characterization result

Other elements detected in significant amounts were silicon (Si) at 14.6% and aluminum (Al) at 2.9%, with their respective oxides being SiO_2 (24.0%) and Al_2O_3 (4.3%). The presence of Si and Al is characteristic of montmorillonite as a layered clay mineral rich in silica and alumina, indicating that the montmorillonite phase remains present in the composite after modification with ZnO and is likely to continue functioning as a high-surface-area supporting matrix for the adsorption process.

The presence of other elements and oxides such as Fe_2O_3 (2.22%), ZrO_2 (2.5%), and Yb_2O_3 (0.2%) in small amounts also has the potential to contribute to improving the composite's performance. Fe_2O_3 is known to absorb light in the visible region and act as an electron acceptor, thereby reducing the recombination rate of electron–hole pairs (Mishra & Chun, 2015). Meanwhile, ZrO_2 can enhance thermal stability and strengthen structural bonding (Lee et al., 2025), while Yb^{3+} doping has been reported to modify the energy band structure of ZnO (Chaudhary et al., 2022), thus extending its spectral response toward visible light.

Photocatalytic Degradation

The photodegradation of methylene blue with varying masses of the ZnO/MMT composite was analyzed using a visible light spectrophotometer. In

this experiment, the composite masses used were 0.05 g, 0.1 g, 0.15 g, 0.2 g, and 0.25 g, tested under various conditions, including the use of montmorillonite + ZnO (MMT/ZnO) under UV light, ZnO under UV, and montmorillonite under UV. The maximum wavelength was first determined within the range of 400–700 nm, resulting in a maximum wavelength of 665 nm. The results of the visible spectrophotometric analysis are presented in Figure 4.

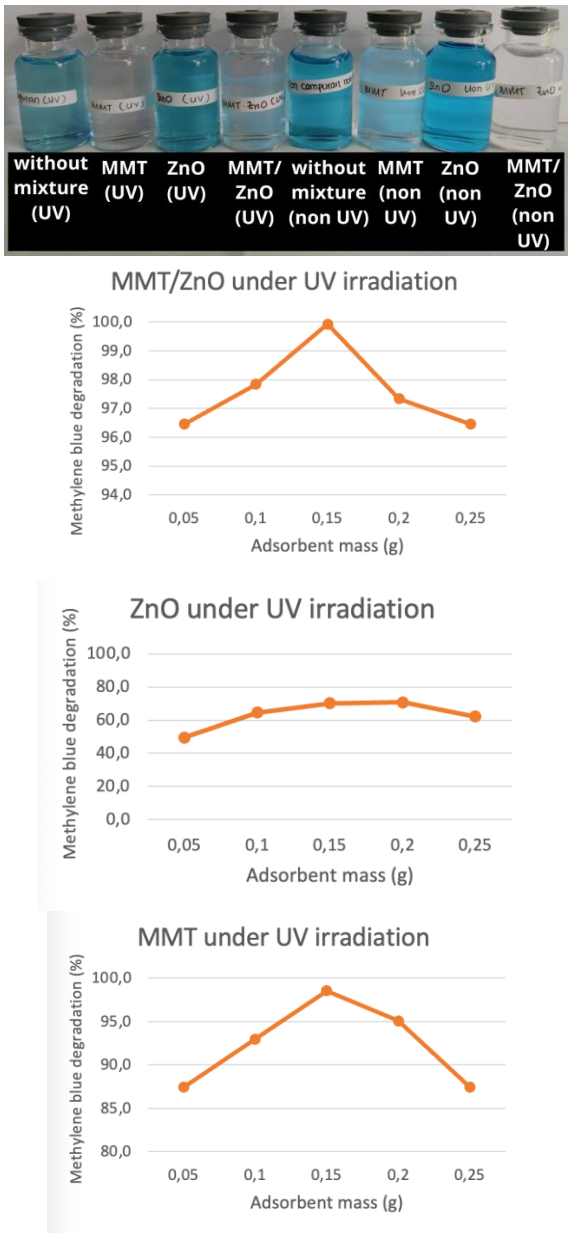

Figure 4. Effect of adsorbent mass on methylene blue degradation under UV irradiation. The plotted values represent processed data (percentage degradation of methylene blue) for (a) MMT/ZnO composite, (b) ZnO, and (c) MMT.

The variation in the mass of the ZnO/MMT composite in the range of 0.05 g to 0.25 g showed the influence of mass changes on the amount of methylene blue adsorbed. However, the data showed that masses of 0.1 g, 0.15 g, and 0.2 g were more effective in removing methylene blue compared to the other masses. Therefore, these three masses were selected as the optimal variations for adsorption testing without UV, such as the use of montmorillonite + ZnO (MMT/ZnO) without UV light, ZnO without UV, and montmorillonite without UV, and the results of the visible spectrophotometric test can be seen in Figure 5. The selection of these masses was intended to obtain data that describes the adsorption mechanism of the composite optimally. In addition, this non-UV variation was also used for comparative analysis with the photocatalytic degradation process carried out at the same mass range under UV irradiation. These results confirm the presence of synergy between the adsorption capability of montmorillonite and the photocatalytic activity of ZnO, which is the key factor in increasing the degradation efficiency of MB and distinguishes this study from previous studies.

Based on the adsorption tests of methylene blue by the MMT/ZnO composite as well as by the single MMT and ZnO materials without UV irradiation, the results showed an efficiency enhancement pattern influenced by the variation in adsorbent mass. The measurement results indicated that adsorption efficiency increased with increasing adsorbent mass; however, for MMT/ZnO and MMT without UV, the efficiency peaked at 0.15 g after rising from 0.1 g, followed by a slight decrease at 0.2 g. This decline suggests the possible agglomeration of particles and a reduction in effective surface area, thereby hindering the contact between methylene blue molecules and the active sites of the adsorbent. This observation is consistent with the findings of Sasikala et al. (2019), which explained that the decrease in efficiency at higher adsorbent masses (≥ 0.2 g) may be caused by particle agglomeration that covers the active pores, thereby reducing the effective surface area for the contact between methylene blue molecules and the active sites Sasikala et al., 2019). This phenomenon is consistent with the principle of heterogeneous photocatalysis, where the reaction occurs only on the active surface of the catalyst exposed to light. When particles stick together, the distribution of UV light becomes uneven, causing a decrease in the number of electron-hole pairs formed (J. Jiang et al., 2017). As a result, the formation of hydroxyl radicals (•OH) and superoxide ($O_2^{\bullet -}$) as the main oxidizing species in the

photodegradation process also decreases (Pannak et al., 2018). Thus, the degradation efficiency decreases not only due to the reduction in adsorption capacity but also due to the decrease in photocatalytic activity caused by the reduced excitation on the surface of ZnO/MMT.

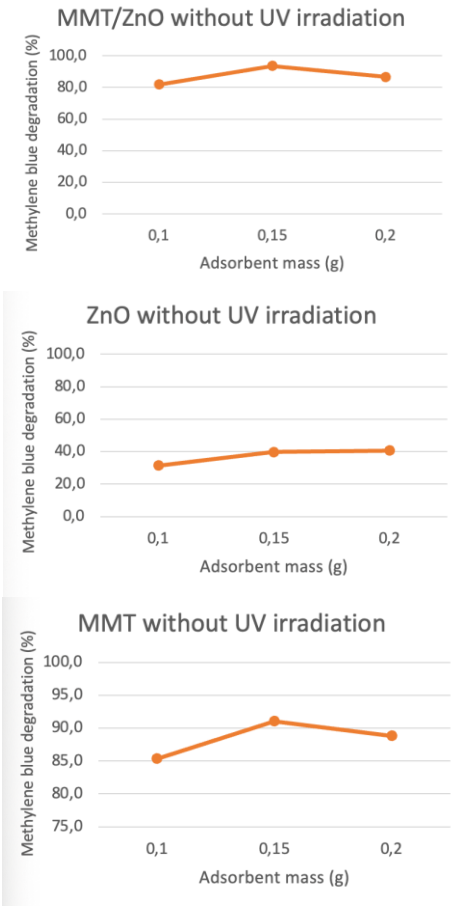

Figure 5. Effect of adsorbent mass on methylene blue degradation in the absence of UV irradiation. The plotted values represent processed data (percentage degradation of methylene blue) for (a) MMT/ZnO composite, (b) ZnO, and (c) MMT.

According to Latupeirissa et al. (2018), the surface area and the interconnected pore structure (macro, micro, and transitional) play an important role in the efficiency of methylene blue adsorption. Adsorption following the Freundlich isotherm indicates that the adsorbent surface is heterogeneous and has many active sites for dye molecule adsorption. Furthermore, to determine how much methylene blue was degraded, a comparison between UV and non-UV conditions was conducted, as shown in Figure 6.

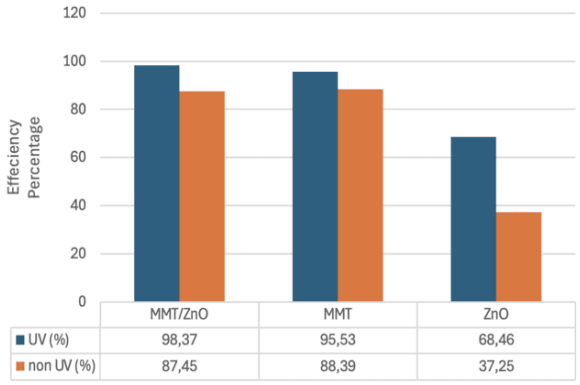

Figure 6. Methylene Blue Degradation

Based on the results obtained, MMT/ZnO under UV irradiation showed a percentage efficiency of 98.37%. This high efficiency was followed by MMT under UV at 95.53% and MMT without UV at 88.39%. Meanwhile, ZnO showed lower efficiency, namely 68.46% under UV conditions and 37.25% under non-UV conditions. The difference between the UV and non-UV treatments indicates that the photocatalytic degradation contribution varies among the materials.

The photocatalytic contribution to degradation can be calculated based on the difference in efficiency between the material with UV and without UV. The results obtained showed that the highest photocatalytic contribution was found in ZnO, namely 31.21%, followed by MMT/ZnO at 10.92%, and MMT at 7.14%. This indicates that ZnO has higher photocatalytic activity compared to the other two materials. This capability is in line with the findings of Raganata et al. (2019) that ZnO nanoparticles act as an effective photocatalyst in the degradation of methylene blue, with an increase in the percentage of degradation. ZnO is known to have an energy gap of 3.37 eV so that it can generate electron–hole pairs effectively when excited by UV light, producing hydroxyl radicals (•OH), which are strong oxidizing agents for degrading organic compounds such as methylene blue.

The MMT/ZnO composite exhibited the highest overall efficiency, indicating a synergy between the adsorption capability of MMT and the photocatalytic activity of ZnO. Adsorption played a dominant role in the MMT/ZnO system, in which 87.45% of the removal was derived from pure adsorption. The photocatalytic degradation in this system contributed only 10.92%, but this contribution was still significant in enhancing the overall efficiency, as shown in Table 1.

Table 1. Degradation efficiency and photocatalytic contribution

Sampel	Condition	Efficiency (%)	Photocatalytic (%)
MMT/ZnO	UV	98,37	10,92
MMT/ZnO	Non-UV	87,45	-
MMT	UV	95,53	7,14
MMT	Non-UV	88,39	-
ZnO	UV	68,46	31,21
ZnO	Non-UV	37,25	-

These results are consistent with the study of Zango et al. (2022), which stated that MMT modified with metal or metal oxide particles showed a significant increase in adsorption capacity and photocatalytic activity toward organic contaminants, including industrial dyes. In addition, the findings of Mustapha et al. (2020) reported that the porous structure and polar network of ZnO in the kaolin/ZnO composite enhanced the diffusion of pollutants onto the catalyst surface and prevented particle agglomeration, resulting in a larger active surface area as well as higher adsorption and degradation efficiency. Yu et al. (2021) stated that although ZnO possesses high photocatalytic activity, its combination with porous adsorbents such as biochar can enhance the overall efficiency of the system by increasing adsorption capacity and facilitating photogenerated electron transfer.

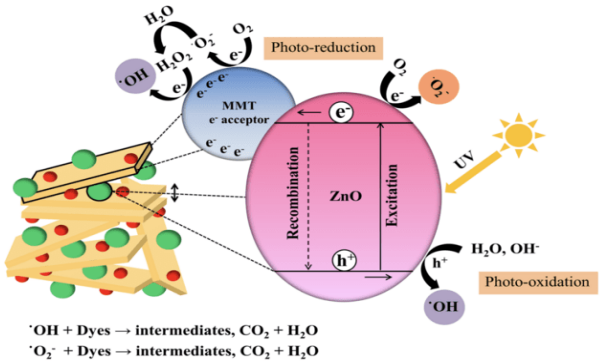

Figure 7. Photodegradation mechanism (Pannak et al., 2018)

The mechanism of photodegradation is illustrated in Figure 7. Initially, electron–hole (e^-–h^+) pairs are separated under UV-C irradiation, where electrons are excited to the conduction band, and holes remain in the valence band of nanosized ZnO. In the presence of MMT clay, the photogenerated electrons are transferred to the vacant metal d-orbitals within the MMT galleries (Hassani et al., 2017). This electron transfer process facilitates the separation of e^-–h^+ pairs. Subsequently, the electrons trapped in MMT can react with O_2 adsorbed on the photocatalyst surface to produce active species such as O_2^-, •OH, and H_2O_2. At the same time, the positive holes in the valence band react with H_2O to generate •OH radicals. All the generated radicals are considered the main active species responsible for degrading the organic dye molecules (Pannak et al., 2018).

CONCLUSION

A montmorillonite–ZnO (MMT/ZnO) composite was successfully synthesized and characterized, confirming the coexistence of ZnO and montmorillonite phases and enabling a dual function of adsorption and photocatalysis for methylene blue removal. Under UV irradiation, the MMT/ZnO composite exhibited the highest overall degradation efficiency (~98%), while in the absence of UV, it still removed more than ~87% of methylene blue, indicating that adsorption by montmorillonite is the dominant pathway. In comparison, single ZnO showed lower overall efficiency but a higher relative photocatalytic contribution, whereas single MMT showed strong adsorption with a weaker photocatalytic response. These results demonstrate a synergistic interaction in the MMT/ZnO system, where the montmorillonite matrix concentrates dye molecules near ZnO active sites and ZnO photocatalysis enhances the final removal efficiency.

Given its high performance, use of inexpensive starting materials, and relatively simple synthesis route, the MMT/ZnO composite is a promising candidate for the treatment of dye-containing wastewater, particularly from the textile industry. Future studies should address the long-term stability and regenerability of the composite over multiple cycles, its performance in continuous-flow or real wastewater systems, and the influence of coexisting ions and organic pollutants on its adsorption–photocatalytic behavior.

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