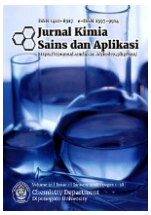
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Adsorption of Methyl Red Dye on Fixed-Bed Column Using Bottom Ash Adsorbent from Palm Oil Mill Waste

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Abstract

Methyl red is an azo compound widely applied in the textile industry as a fabric dye. Methyl red is very difficult to decompose because it is stable to light, temperature, and oxidation. One of the simplest and low-cost methods is adsorption. This study aims to determine the characteristics of bottom ash as an adsorbent and analyze the adsorption process with a fixed-bed column system. The research was conducted by varying the flow rate of 5, 10, and 15 L/min, adsorbent bed height of 4, 8, and 12 cm, and contact time of 60, 120, and 180 minutes. The highest removal efficiency, 93%, was achieved at a contact time of 180 minutes, a flow rate of 5 L/min, and a bed height of 12 cm. The physical properties of the bottom ash adsorbent include a pore volume of 0.0032 cm^3/g, a specific surface area of 89.801 m^2/g, and an average pore size of 58.523 nm. The maximum adsorption capacity was found to be 0.850 mg/g. The adsorption process of methyl red onto bottom ash follows the Langmuir isotherm model.

1. Introduction

The largest manufacturing industry in Indonesia is currently the textile industry, which has seen significant growth due to its promising prospects and national priority status [1]. The textile production process, which involves multiple stages, generates various types of waste, including solid, liquid, and gaseous waste. In particular, liquid waste from the dyeing process contains high ammonia levels, posing a risk of water pollution. One of the commonly used dyes in the textile industry is methyl red (MR) [2, 3].

Most textile dyes are derived from azo compounds and benzene derivatives, which are highly resistant to degradation. Textile mills consume large amounts of water in processes such as bleaching, dyeing, and washing, often discharging untreated wastewater into water bodies. This wastewater primarily contains organic dyes that disperse well in water, making their removal challenging using conventional methods. The presence of these dyes alters water coloration, negatively impacting the visual landscape [4].

Additionally, high dye concentrations reduce light penetration into aquatic environments, lowering photosynthesis rates and decreasing oxygen levels essential for aquatic flora and fauna. Many organic dyes exhibit toxic, mutagenic, and carcinogenic properties. In developing countries, dye-contaminated water is often used for irrigation, further spreading pollution. These dyes contaminate irrigation water and disrupt soil microbial activity, negatively impacting soil health and fertility [5].

Various methods have been employed to treat textile industry effluents and mitigate environmental pollution. Common wastewater treatment techniques include electrocoagulation, chemical oxidation, ozonation, membrane degradation, and filtration. Among these, adsorption is widely recognized as an effective, simple, and cost-efficient technique [1, 3, 6, 7, 8].

The effectiveness of adsorption largely depends on the choice of adsorbent, as it influences the purification process [5, 8, 9, 10, 11]. A key characteristic of an effective adsorbent is its surface area, as a larger surface area enhances adsorption capacity by providing more active

sites for adsorbate interaction [7, 8]. Various adsorbents have been studied for MR removal, including activated carbon, zeolite, mesoporous silica, and biomass-based materials.

Biomass waste-derived adsorbents, such as oil palm waste (e.g., palm shells and fibers), are commonly preferred for commercial applications. Many factories use palm shells and fibers as boiler fuel, producing bottom ash as a byproduct. Bottom ash consists primarily of silica (SiO_2) and alumina (Al_2O_3), the main components of zeolite. Since zeolites are known for their MR adsorption capacity, bottom ash, with a similar composition, also has the potential to act as an effective adsorbent for MR removal [11, 12, 13, 14].

Research on bottom ash-based adsorbents has primarily focused on heavy metal adsorption, with limited studies on MR adsorption. Currently, bottom ash remains underutilized and poorly managed, posing a challenge for the palm oil industry due to its increasing accumulation and the large storage areas required [15]. Malviya *et al.* [16] and Savcı and Uysal [17] have explored the use of bottom ash for methyl orange adsorption. Utilizing bottom ash as an adsorbent presents a sustainable approach to mitigating environmental pollution [8, 15, 18].

In the palm oil industry, bottom ash from combustion residues is often left in accumulated piles, leading to potential groundwater contamination [19]. Consequently, various innovations have been developed to repurpose bottom ash, including its use as a cement substitute and an adsorbent. This study investigates the potential of bottom ash as an adsorbent for MR removal using a column system, following an initial characterization of its properties.

2. Experimental

2.1. Materials and Equipment

This adsorption study utilized bottom ash obtained from PT IBAS palm oil mill in North Aceh Regency, along with distilled water, methyl red ($C_{15}H_{15}N_3O_2$, 99% Merck), and tapioca starch. The equipment used in this research was an acrylic adsorption column with a diameter of 6 cm and a height of 30 cm, scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDX, CARL ZEISS EVO MA 10), X-ray diffraction (XRD, Shimadzu XRD-6100), Fourier transform infrared (FTIR) spectroscopy (IR Prestige 21), oven (Memmert UN 30), UV-Vis Spectrophotometer (UV-1800), and a surface area analyzer.

2.2. Adsorbent and Adsorbate Preparation

The preparation of the bottom ash adsorbent began by weighing 100 g of bottom ash, which was then sieved using a 20-mesh sieve. An adhesive was prepared by mixing 15 g of tapioca starch with 45 mL of distilled water. The bottom ash was then mixed with the adhesive until a homogeneous mixture was formed and placed into an iron mold with a diameter of 1.5 cm and a height of 1 cm, representing a laboratory-scale process. The molded samples were oven-dried at 105°C until a constant weight was achieved. After drying, the adsorbent was removed from the mold and characterized using BET surface area analysis, SEM/EDX, XRD, and FTIR spectroscopy. The adsorbate solution was prepared by dissolving 20 mg of MR powder in a 1000 mL volumetric flask filled with distilled water to obtain a final concentration of 20 ppm.

2.3. Adsorption Process using Column

The adsorption process was conducted using an acrylic adsorption column with a diameter of 6 cm and a height of 30 cm. In the initial stage, the pre-formed and dried adsorbent was placed into the column, with bed heights varied at 4, 8, and 12 cm. Flow rates were adjusted to 5, 10, and 15 L/min, and adsorption times were set at 60, 120, and 180 minutes. A 20 ppm MR solution was introduced into the column in an up-flow manner, as illustrated in Figure 1. Samples were collected from the column via a valve at predetermined contact times and subsequently analyzed using a UV-Vis spectrophotometer. Table 1 presents the research variables.

The efficiency and adsorption capacity were calculated using Equations (1) and (2).

$$Removal\ efficiency\ (\%) = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

$$q_t = \frac{V\,(C_0 - C_e)}{m} \quad (2)$$

Where, C_0 is the initial concentration of MR solution ($mg.L^{-1}$), C_e is the final concentration of MR solution ($mg.L^{-1}$), q_t is the adsorption capacity at time t ($mg.g^{-1}$), m is the adsorbent mass (g), V is volume of MR solution (L).

Table 1. Variables in research

Variable	Value
Bed heights, cm	4, 8, 12
Flow rates, L/min	5, 10, 15
MR concentration, ppm	20
Contact time variations, min	60, 120, 180

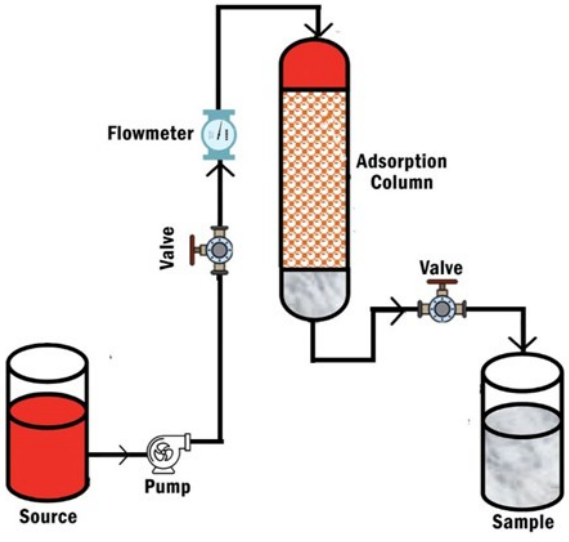


Figure 1. Schematic of the MR adsorption process using a bottom ash adsorbent in a column system

3. Results and Discussion

3.1. Adsorbent Characterization

The results of the bottom ash surface area analysis are shown in Table 2. The data indicate a relatively large surface area of 89.98 m^2/g, which enhances its adsorption capacity by providing more active sites for adsorption [20]. Figure 2 shows the structure and surface morphology of the adsorbent, analyzed by SEM at 500× magnification, with EDX used for elemental composition (Table 3). The results indicate that C, O, and Si are the dominant elements in the adsorbent, suggesting good adsorption potential due to their ability to interact with the dye's chemical structure and create favorable adsorption conditions. These elements contribute to adsorption through mechanisms such as electrostatic forces, hydrogen bonding, and hydrophobic interactions, all aiding MR uptake.

Figure 2a shows the SEM image of bottom ash before adsorption, revealing an average pore size of approximately 10 µm. In contrast, Figure 2b shows the adsorbent after adsorption, where the pores appear covered and more compact due to MR adsorption, indicating that the adsorbate has filled and blocked the pore spaces. BET analysis shows that the adsorbent derived from palm oil mill bottom ash has pores larger than 50 nm. Based on this pore size observed in the SEM analysis, the adsorbent is classified as a macroporous material. This classification aligns with other literature, which reports that bottom ash contains three types of pores: macropores (> 55 nm), mesopores (7–8 nm), and micropores (< 1 nm) [21, 22, 23].

The adsorbent was also identified using FTIR spectroscopy, with spectra recorded before and after adsorption, as shown in Figure 3. Identification is seen from the peaks that appear from several functional groups. Before adsorption, a peak at 958–954 cm^{-1} corresponds to the Si–O–Si bond, indicating the presence of silica in the bottom ash [21]. Another peak at 812 cm^{-1} represents the Al–O–Al bond, characteristic of alumina. Additionally, peaks at 1629–1504 cm^{-1} correspond to the bending vibrations of the –OH group, while the peak at 1696 cm^{-1} is attributed to C=O stretching.

Table 2. Physical properties of the bottom ash adsorbent

Sample	Pore volume (cm^3/g)	Specific surface area (m^2/g)	Pore size (nm)
Bottom ash	0.0032	89.801	58.523

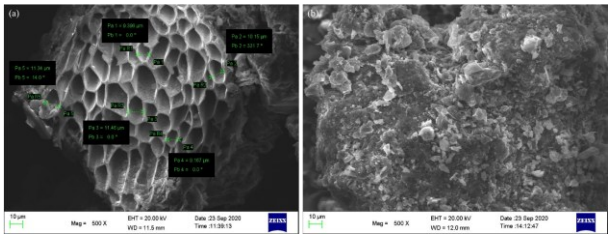


Figure 2. SEM images of the adsorbent (a) before and (b) after MR adsorption

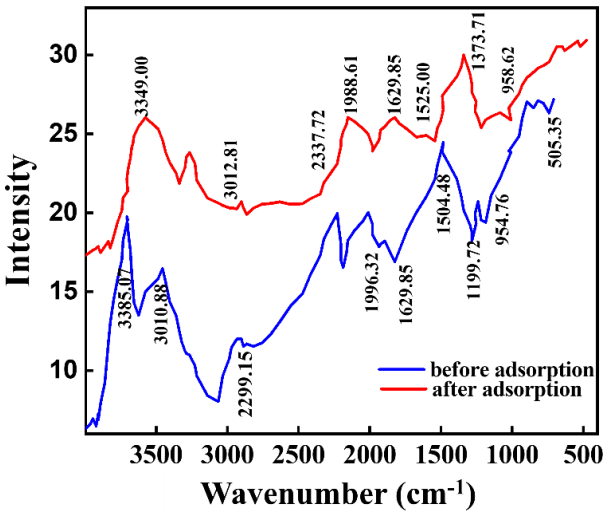


Figure 3. FTIR spectra of bottom ash before and after adsorption

Table 3. Physical properties of the bottom ash adsorbent

No	Element	Atom (%)	
		Before adsorption	After adsorption
1	C	45.77	33.63
2	O	36.9	45.13
3	Mg	1.01	0.95
4	Al	0.36	4.63
5	Si	11.88	8.63
6	P	0.55	0.3
7	K	1.88	4.04
8	Ca	1.36	2.05
9	Fe	0.28	0.64

After adsorption, spectral changes were observed. A peak at 3556 cm^{-1} corresponds to hydrogen bonding and hydroxyl groups, while stretches at 3468–3404 cm^{-1} indicate N–H stretching from aromatic primary amines. The peak at 3349 cm^{-1} is associated with azo bonds, while the peak at 1525 cm^{-1} corresponds to N=N stretching. Additionally, a peak at 1369 cm^{-1} represents the C–N bond of aromatic amines, and the peak at 1373.71 cm^{-1} is attributed to CH_3 and CH_2 bonding. These findings are consistent with previous FTIR analyses of MR adsorption on bottom ash, confirming successful adsorption [24].

3.2. Effects of Flow Rate, Contact Time, and Bed Height on MR Adsorption Efficiency and Capacity

Figure 4 illustrates the significant impact of MR flow rate and bed height on adsorption efficiency. A higher flow rate leads to lower adsorption efficiency, as the reduced contact time between MR and the adsorbent limits the adsorption process. Conversely, a lower flow rate allows for prolonged interaction, enhancing adsorption efficiency. As shown in Figure 6, the highest adsorption efficiency of 93% was achieved at a flow rate of 5 L/min and a bed height of 12 cm. This result is slightly different from the result obtained by Hamzah *et al.* [20], which is 98%.

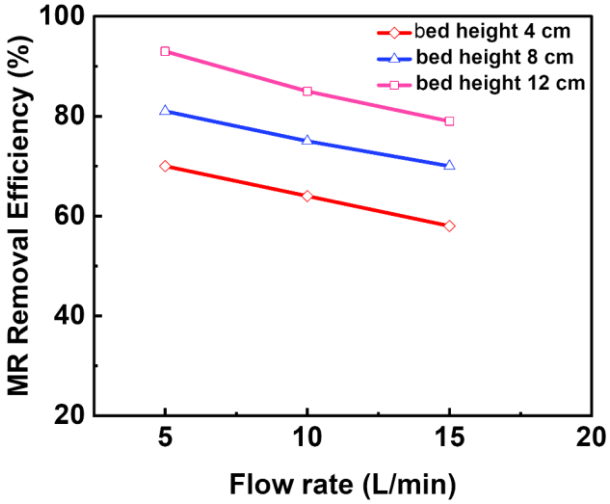


Figure 4. The effect of flow rate on the removal efficiency of MR

Figure 5 shows the relationship between contact time and separation efficiency, with adsorption durations from 60 to 180 minutes. The optimal contact time was observed at 180 minutes, achieving a maximum removal efficiency of 93% at a bed height of 12 cm. Contact time is a crucial factor in adsorption, as it determines the extent of interaction between the adsorbate and adsorbent. According to reaction rate theory, the reaction rate depends on the number of collisions occurring per unit of time [23]. Longer contact times increase collisions, driving the reaction toward equilibrium [24, 25]. The time required to reach adsorption equilibrium varies with the nature of adsorbent–adsorbate interactions.

The relationship between adsorption capacity and contact time is shown in Figure 6. Adsorption capacity increases with bed height due to the larger surface area and the availability of more active sites. At higher bed heights, a greater adsorbent surface area enhances MR adsorption capacity. The highest adsorption capacity of 0.850 mg/g was achieved at a contact time of 180 min and a bed height of 12 cm [11, 26, 27, 28, 29]. As contact time increases, more adsorbate accumulates on the adsorbent surface until equilibrium is reached. However, excessive contact time can lead to adsorbent saturation, preventing further adsorption and causing the release of adsorbate.

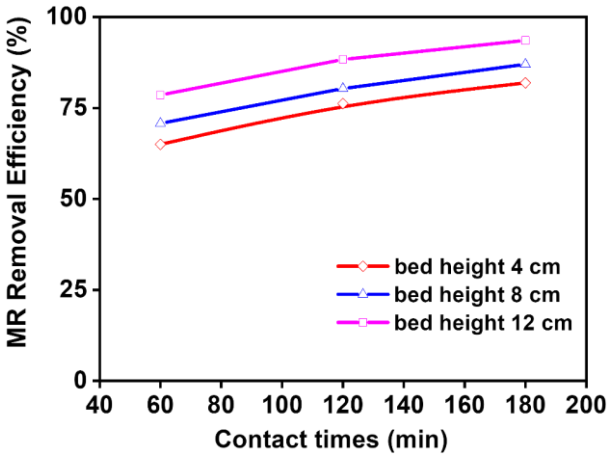


Figure 5. Effect of contact time and MR adsorption efficiency at different bed heights

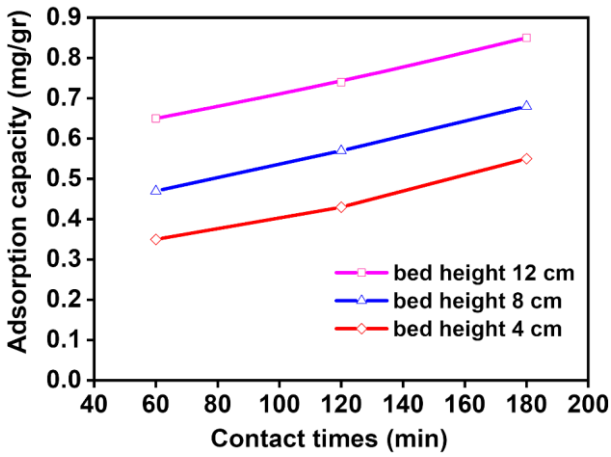


Figure 6. Effect of contact time and MR adsorption capacity at different bed heights

3.3. Adsorption Isotherm

Langmuir and Freundlich models were used to represent the adsorption equilibrium data in this study. The Langmuir model assumes that the adsorbent surface is homogeneous, with constant adsorption energy over the entire surface. This model also assumes that adsorption is localized, and each location can only absorb one molecule or atom, while the Freundlich adsorption isotherm model assumes that the adsorbate binding process occurs on heterogeneous surfaces [2, 13, 21, 28]. The isotherm models suitable for the equilibrium curves must be determined to optimize the sorption system design for MR sorption on bottom ash. In this study, the predicted values of the model and experimental data were validated by comparing the experimental adsorption capacity with the adsorption capacity estimated by this model through the coefficient of determination (R^2, a value close to or equal to 1 in Table 4). The MR adsorption isotherm model on bottom ash follows the Langmuir model shown in Figure 7 [8, 23, 26, 28].

Table 4. Langmuir and Freundlich isotherm parameters for MR adsorption onto bottom ash

Isotherm type	Parameter	Value	Equation form
Langmuir	q_m	0.889	$q = \frac{q_m K_L C_e}{1 + k_L C_e}$
	k_L	0.044	
	R^2	0.998	Non-linear
Freundlich	k_F	0.519	$q = k_f C_e^{\frac{1}{n}}$
	n	-1.812	
	R^2	0.815	Non-linear

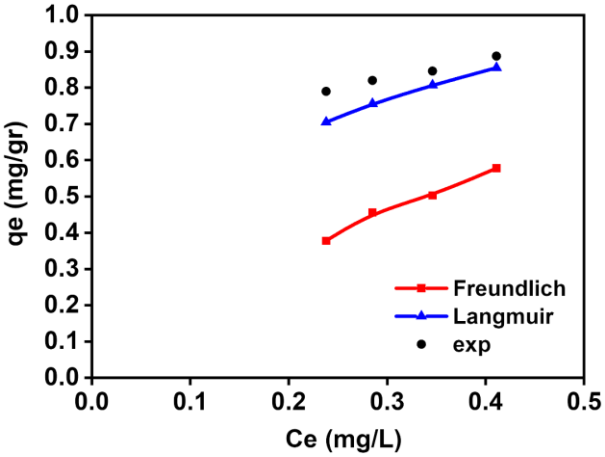


Figure 7. Adsorption isotherm of MR onto bottom ash

4. Conclusion

The research was conducted by varying the flow rate (5, 10, and 15 L/min), adsorbent bed height (4, 8, and 12 cm), and contact time (60, 120, and 180 minutes). The results showed that the highest adsorption efficiency, 93%, was achieved at a contact time of 180 minutes, a flow rate of 5 L/min, and a bed height of 12 cm. The highest adsorption capacity, 0.850 mg/g, was obtained at a bed height of 12 cm and a contact time of 180 minutes. The adsorption of MR using bottom ash follows the Langmuir isotherm model, as evidenced by the strong agreement between the model and experimental data, with an R^2 value of 0.998. These results indicate that bottom ash is a promising adsorbent for MR removal. Moreover, utilizing waste materials such as bottom ash offers a cost-effective source of raw materials and contributes to sustainable waste management solutions.

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