



Sustainable Methylene Blue Removal using NaOH-Activated Rice Husk Charcoal

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Abstract. *Water pollution from synthetic dyes such as methylene blue (MB) remains a serious environmental issue, particularly from textile and paper industries. This study explores rice husks as a low-cost bioadsorbent through four treatments: raw husks (SM), NaOH-activated husks (SA), rice husk charcoal (A), and NaOH-activated charcoal (AA). For the activated materials, the rice husks were immersed in a 6 M NaOH solution for 4 hours with intermittent manual stirring, followed by washing until neutral pH was reached and drying. Adsorption efficiency was evaluated using UV-Vis spectroscopy, while FTIR identified functional groups. AA demonstrated the best performance, reaching 95.28% efficiency at 6 hours and 96.96% at 18 hours, with equilibrium achieved at 6 hours. This enhanced adsorption is attributed to π - π stacking between aromatic structures and MB molecules, as well as electrostatic interactions from negatively charged $-OH$ groups introduced by alkaline activation. In addition to its excellent adsorption capability, the application of rice husks as a renewable bioresource offers clear environmental and economic benefits through waste conversion and reduced dependence on conventional activated carbon. These results confirm the potential of NaOH-activated rice husk charcoal as an efficient, sustainable, and environmentally friendly adsorbent for dye wastewater treatment.*

Keywords: Methylene Blue, Rice Husk, Activated Charcoal, Bioadsorbent, UV-Vis Spectrometer, FTIR Spectrometer, Wastewater Treatment

Introduction

Water pollution caused by synthetic dyes such as methylene blue (MB) is a major environmental concern, particularly originating from the textile, paper, and dye industries [1–3]. These wastewaters often contain high concentrations of stable dyes that reduce light penetration and negatively affect aquatic ecosystems [4, 5]. This situation highlights the need for wastewater treatment technologies that are effective, cost-efficient, and sustainable.

One of the most widely employed approaches for dye removal is adsorption, a process in which dissolved substances are captured using porous materials. This method is particularly attractive because it is simple, cost-effective, and highly efficient in reducing dye concentrations in wastewater [6]. Among the potential adsorbents, activated carbon derived from agricultural residues such as rice husks presents a promising option. Rice husks are rich in silica, cellulose, and lignin, which, after suitable modification, can be transformed into adsorbent materials with high surface activity [7, 8]. Their abundant availability in Indonesia further enhances their value as an inexpensive and easily accessible raw material. Previous studies have confirmed the



effectiveness of rice husks in adsorbing MB. Nevertheless, systematic investigations that compare different treatment stages of rice husks including raw material, chemical activation, and the combined processes of carbonization and activation, remain limited. Such studies are essential to determine the most favorable conditions for optimizing the adsorption process [9-11].

NaOH was chosen as the activating agent over other chemicals, such as KOH, due to several technical and practical advantages. Activation with NaOH is generally more cost-effective and can produce carbon materials with high surface area in a relatively short time, especially when using agricultural residues like rice husks [12-14]. Moreover, NaOH activation tends to result in lower ash content, enhancing the purity of the final carbon product. The chemical properties of Na⁺ ions also promote a more uniform distribution of oxygen-containing functional groups, which improves adsorption performance [13, 14]. These characteristics make NaOH a more efficient, practical, and sustainable choice for large-scale bio-adsorbent production compared to KOH [12, 13].

Accordingly, this study holds considerable urgency as it addresses a knowledge gap regarding the effectiveness of different rice husk treatments as adsorbents. The results of this work are expected to provide a basis for selecting the most efficient and cost-effective method for potential application at an industrial scale. Beyond its practical relevance for reducing pollution in industrial sectors, this research also contributes to the protection of aquatic ecosystems while enhancing the added value of agricultural waste. These outcomes are in line with the objectives of the Sustainable Development Goals (SDGs), particularly those related to clean water, sanitation, and environmentally sound waste management.

Materials and Methods

Materials

In this study, two types of rice husk-based materials were employed. The first was raw rice husks, collected directly from a rice mill in Bandung Regency, while the second was rice husk charcoal, obtained through purchase from an e-commerce platform based in Bandung. The rice husk charcoal was produced via combustion and was characterized by its predominantly black color (approximately 99%). A visual representation of the rice husks used in this study is provided in **Figure 1**. Other materials in this study include NaOH as the activating agent, citric acid as a neutralizing agent during post-activation washing, and methylene blue as the dye used for adsorption tests. The samples investigated comprised four variations: raw rice husks (SM), NaOH-activated rice husks (SA), rice husk charcoal (A), and NaOH-activated rice husk charcoal (AA).

Synthesis Method

In the subsequent stage, both raw rice husks and rice husk charcoal designated for activation were immersed in a 6 M NaOH solution for 4 hours. During immersion, manual stirring was carried out for approximately 1 minute at each hourly interval. The activation process was conducted with a mass ratio of rice husk to NaOH of 1:1. To control possible side reactions and ensure proper removal of residual alkali, the samples were thoroughly washed with water until a neutral pH was achieved. Subsequently, they were dried in an oven at 110 °C for 2.5 hours. The activated

products obtained were subsequently used as bioadsorbents to evaluate their adsorption performance.



Figure 1. Basic Materials of the Samples: (a) Raw Rice Husk and (b) Rice Husk Charcoal.

Experimental Method

To determine the adsorption capacity of rice husk-based adsorbents toward methylene blue (MB), the four types of bioadsorbents were immersed in a MB solution with a concentration of 5 mg/L. The methylene blue used in this study had a purity of $\geq 99.0\%$. A ratio of 1 g bioadsorbent to 50 mL MB solution (1:50 w/v) was used during the immersion tests. Adsorption efficiency was evaluated at various contact times of 3, 6, 9, 12, 15, and 18 hours by analyzing the solutions obtained at each interval. All samples were immersed in MB solutions maintained at neutral pH and room temperature.

Characterization Method

Characterization in this study was conducted using two techniques: UV-Vis spectrophotometry to evaluate adsorption efficiency and Fourier Transform Infrared (FTIR) spectroscopy to examine the functional groups of the bioadsorbents. FTIR analysis was carried out on four types of untreated samples, allowing the identification of functional group variations resulting from different treatments before adsorption. Meanwhile, UV-Vis spectrophotometry was applied to all four bioadsorbents across six contact time intervals, enabling quantitative determination of MB removal efficiency. In total, 24 samples were analyzed to provide a comprehensive assessment of the adsorption performance. The adsorption efficiency (E) was determined using the equation:

$$E = \frac{T_s}{T_{dw}} \times 100\%$$

where T_s and T_{dw} represent the transmittance of the samples and transmittance of distilled water.

Results and Discussion

UV-Vis Analysis

This study utilized four types of samples: raw rice husks (SM), NaOH-activated rice husks (SA), rice husk charcoal (A), and NaOH-activated rice husk charcoal (AA). Each sample was immersed in an MB solution with contact times of 3, 6, 9, 12, 15, and 18 hours. The coding details are presented in Table 1, for example SM3h refers to raw rice husks immersed for 3 hours, while AA6h denotes NaOH-activated rice husk charcoal immersed in the MB solution for 6 hours, respectively.

Table 1. Tested Rice Husk Samples

Sample Code	Sample Type	Treatment	Tested Sample
SM	Raw Rice Husks	Raw rice husk was washed and then dried	SM3h, SM6h, SM9h, SM12h, SM15h, SM18h
SA	NaOH-activated Rice Husks	Raw rice husk was washed, activated with 6M NaOH, and then dried	SA3h, SA6h, SA9h, SA12h, SA15h, SA18h
A	Rice Husk Charcoal	Rice husk charcoal was washed and then dried	A3h, A6h, A9h, A12h, A15h, A18h
AA	NaOH-activated Rice Husk Charcoal	Rice husk charcoal was washed, activated with 6M NaOH, and then dried	AA3h, AA6h, AA9h, AA12h, AA15h, AA18h

The visual observations from the adsorption tests revealed significant differences among the samples, as shown in **Figure 2**. In the case of NaOH-activated rice husk charcoal (AA), a significant reduction in the intensity of the blue color in the methylene blue solution was already evident after 3 hours of contact. Under this condition, the solution appeared considerably lighter compared to the other samples, indicating that adsorption occurred rapidly during the initial stage. When the contact time reached 6 hours, the blue coloration of the MB solution was almost completely removed, approaching the appearance of pure water, suggesting that adsorption had reached equilibrium at this point.

These results are supported by the UV-Vis characterization of 24 samples, as illustrated in **Figure 3**. The UV-Vis spectrum of the methylene blue (MB) solution exhibited an absorption peak at the maximum wavelength (λ_{max}) of 666 nm. For the raw rice husk (SM) samples, transmittance values

ranged from 48% to 65%, gradually increasing with contact time and reaching the highest value at 18 hours. In contrast, NaOH-activated raw husks (SA) showed lower transmittance levels, ranging from 8% to 40%, with the maximum value also observed at 18 hours. The rice husk charcoal (A) samples exhibited transmittance between 69% and 85%, peaking at 12 hours.

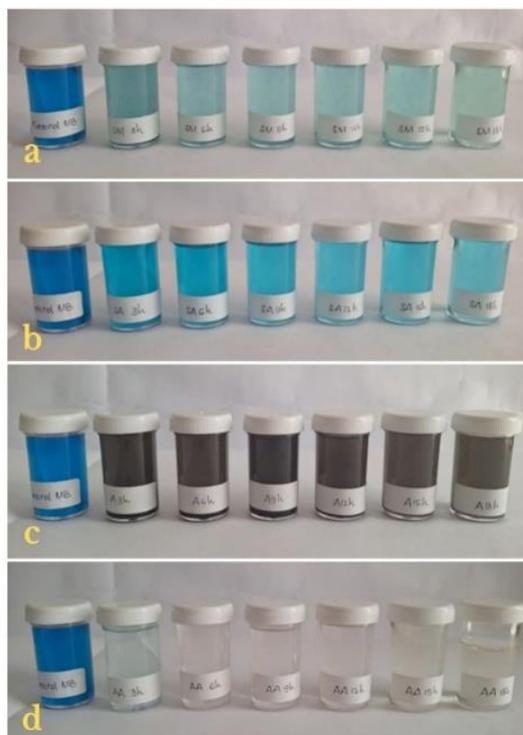


Figure 2. Visual Adsorption of MB Solution Using Bio-Adsorbents: (a) Raw Rice Husk, (b) NaOH-Activated Rice Husk, (c) Rice Husk Charcoal, and (d) NaOH-Activated Rice Husk Charcoal.

However, despite these relatively high transmittance values, the resulting solution appeared darkened due to the presence of residual charcoal particles. A different behavior was observed for NaOH-activated charcoal (AA), where transmittance values were markedly higher, between 87% and 97%, with the maximum recorded at 18 hours. Interestingly, equilibrium appeared to be reached as early as 6 hours, as the transmittance values remained nearly constant (95–97%) up to 18 hours. This result suggests that with only 6 hours of contact, the MB solution had already achieved approximately 95% transmittance and, visually, appeared clear, closely approximating pure distilled water.

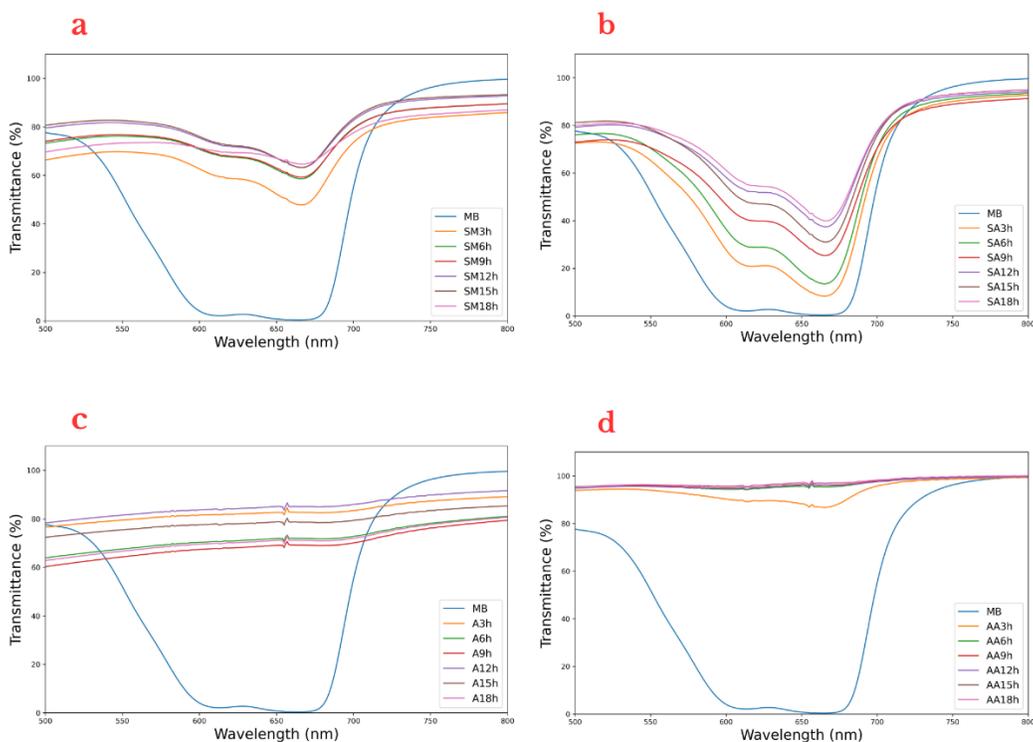


Figure 3. UV-Vis Spectra of MB Solution Adsorbed Using: (a) Raw Rice Husk, (b) NaOH-Activated Rice Husk, (c) Rice Husk Charcoal, and (d) NaOH-Activated Rice Husk Charcoal.

The UV-Vis spectra were used to analyze the adsorption efficiency of each rice husk-based bioadsorbent through a normalization process. The calculated efficiencies were then presented graphically, as shown in **Figure 4**.

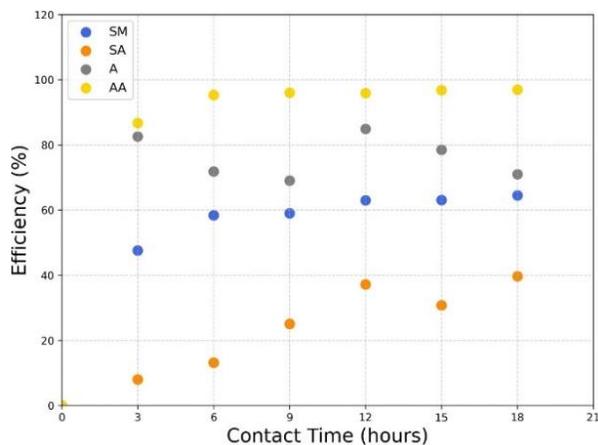


Figure 4. Efficiency at All Contact Time Intervals



The analysis showed that NaOH-activated rice husk charcoal exhibited the highest efficiency among the four samples, reaching 95.277% after only 6 hours of contact and further increasing to a maximum of 96.957% at 18 hours. To facilitate comparison across the samples, the efficiency data were also summarized in tabular form, as presented in Table 2.

Table 2. Adsorption Efficiency of Methylene Blue Solution

Sample Type	Contact Time (h)	Efficiency (%)
SM	3	47.553
	6	58.378
	9	59.01
	12	63.026
	15	63.082
	18	64.461
SA	3	7.986
	6	13.177
	9	25.067
	12	37.159
	15	30.753
	18	39.651
A	3	82.572
	6	71.812
	9	69.032
	12	84.924
	15	78.505
	18	70.967
AA	3	86.683
	6	95.277
	9	96.045
	12	95.879
	15	96.825
	18	96.957

The results of this study demonstrate notable benefits when compared to earlier works. For instance, Hayfron et al. [15] developed zeolite derived from rice husk ash. Although the resulting material exhibited an adsorption efficiency of about 80–90% for methylene blue, the synthesis procedure was relatively complex and required high-temperature treatment. Other studies utilized rice husk as a precursor to produce mesoporous silica nanoparticles [9] and TiO₂–silica nanoparticles [11]. Both materials demonstrated excellent adsorption performance, with efficiencies reaching 95% for mesoporous silica and 96–97% for TiO₂–silica. However, the preparation of such nanoparticle-based adsorbents involved extended and sophisticated synthesis steps, making them less practical for large-scale applications. Similarly, another study reported that rice husk biochar achieved a maximum adsorption efficiency of nearly 99% [16]. Nevertheless, this performance was obtained through pyrolysis, a process that requires an oxygen-free environment and elevated temperatures, thereby presenting similar challenges in terms of complexity and operational demands. Overall, although previous investigations have

successfully achieved high adsorption efficiencies, most remain constrained by costly and labor-intensive synthesis routes that limit practical implementation. Therefore, the present work gains strategic importance, as it emphasizes the development of simpler, more cost-effective bioadsorbent materials with greater potential for real-world wastewater treatment applications.

FTIR Analysis

Differences in the composition of raw materials and the application of activation treatments significantly influence the adsorption performance of rice husk toward methylene blue (MB). In its unmodified form, rice husk contains high levels of lignin, cellulose, hemicellulose, and silica. This composition gives rise to a compact structure with limited porosity, which restricts the availability of active sites for MB adsorption. FTIR analysis of untreated rice husk (**Figure 5**) identified notable absorption bands at 797 cm^{-1} [17], 1080 cm^{-1} [18], and 1155 cm^{-1} [19], corresponding to Si–O vibrations, as well as a broad band near 3400 cm^{-1} that indicates the presence of –OH groups [20]. The dominance of silica-related functionalities explains the relatively poor adsorption capacity of raw rice husk. This interpretation is further supported by UV-Vis measurements at 666 nm , where the MB solution displayed low transmittance following interaction with the unmodified adsorbent [21]. To clarify, the sample peak data are presented in **Table 3**.

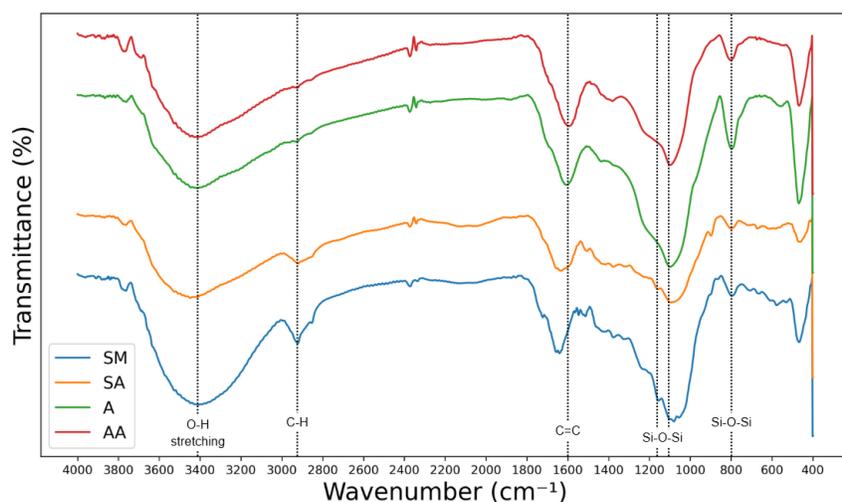


Figure 5. FTIR Spectra of the Samples

The activation of raw rice husk with NaOH reduces part of its silica content, as indicated by the decreased intensity of Si–O absorption bands in the FTIR spectrum. However, since the material was not carbonized, organic components such as cellulose, hemicellulose, and lignin remain dominant and are relatively fragile when exposed to alkaline treatment. This condition leads to a less stable structure, thereby limiting the increase in the number of active sites available for adsorption [22, 23]. As a result, the capacity of NaOH-activated raw husk to remove methylene blue is even lower than that of untreated raw husk. This outcome is further supported by UV-Vis analysis, where the activated sample exhibited the lowest adsorption efficiency among all tested variations.

Table 3. FTIR Peaks of All Samples

SM Peaks (cm ⁻¹)	SM Peaks Transmittance	SA Peaks (cm ⁻¹)	SA Peaks Transmittance	A Peaks (cm ⁻¹)	A Peaks Transmittance	AA Peaks (cm ⁻¹)	AA Peaks Transmittance	Signing
797	89.85	802	93.45	797	73.08	802	87.2	Si-O-Si [17]
1080	27.27	1096	56.50	1099	14.3	1097	35.095	Si-O-Si [18]
1155	37.6	1159	63.1	-	-	-	-	Si-O-Si [19]
1641	61.21	1634	72.24	1604	55.12	1587	54.65	C=C [20]
2922	66.19	2920	75.89	2920	76.75	2924	73.53	C-H [18]
3442	36.43	3449	58.77	3415	53.47	3408	48.9	O-H stretching [21]

Rice husk charcoal exhibited improved adsorption performance compared to untreated husk. This effect arises from the carbonization process, which leads to the decomposition of simple organic groups such as –OH, while simultaneously facilitating the formation of new aromatic rings. These aromatic structures enable π – π stacking interactions with the aromatic rings of methylene blue (MB) molecules. Such interactions are reflected in the FTIR spectrum by the enhanced absorption band observed at around 1604 cm⁻¹, corresponding to aromatic bond vibrations. Moreover, the absorption band at 3415 cm⁻¹, associated with –OH groups, was still observed, indicating that certain active sites remained available to facilitate MB adsorption through electrostatic interactions. These results are consistent with the UV-Vis characterization, which demonstrated a higher transmittance of MB solution compared to raw husk samples, thereby confirming the greater adsorption efficiency [24, 25].

The rice husk charcoal activated with NaOH exhibited the highest adsorption efficiency among all samples. This result can be attributed to the combined effects of carbonization and alkaline activation. The relatively high silica content in rice husks (15–20%) plays a notable role during chemical activation. NaOH reacts with silica (SiO₂) in the husks to form soluble sodium silicate (Na₂SiO₃), which is removed during washing [4, 26, 27]. This reaction generates additional porosity within the carbon framework, enhancing the surface area of the resulting activated carbon. The action of NaOH effectively removed a large fraction of silica, as indicated by the diminished Si–O absorption band near 802 cm⁻¹ and the disappearance of the characteristic Si–O peak around 1150 cm⁻¹. At the same time, the FTIR spectrum revealed a stronger band at 1587 cm⁻¹ associated with aromatic C=C stretching, suggesting an increase in the number of aromatic rings capable of forming π – π stacking interactions with the aromatic structure of methylene blue (MB). Additionally, the observed shift of aromatic bands toward lower wavenumbers points to greater conjugation within the bioadsorbent framework, further supporting stronger π – π interactions. This structural transformation directly translates into a higher adsorption capacity for



MB. Consistent with these findings, UV-Vis analysis showed the most substantial increase in transmittance at 666 nm for this sample. Thus, NaOH-activated rice husk charcoal can be identified as the most efficient adsorbent in this study [24-29].

Conclusions

Based on the results, NaOH-activated rice husk charcoal (AA) was identified as the most effective bioadsorbent for removing methylene blue, achieving more than 95% adsorption efficiency within only 6 hours and reaching nearly 97% after 18 hours. This high performance results from the combined effects of carbonization, which produces an aromatic structure enabling π - π stacking interactions, and alkaline activation, which enhances the number of negatively charged functional groups. Although non-activated charcoal (A) performed better than raw husks, its use may lead to darker solution appearance due to the presence of suspended particles. Therefore, NaOH-activated rice husk charcoal (AA) can be considered a promising option for cost-effective and sustainable treatment of colored wastewater. However, this study did not include BET or SEM analyses, nor pH optimization, which are essential for a more detailed understanding of surface properties and adsorption behavior. Further studies are recommended to perform textural and morphological characterization, adsorption kinetics and isotherm tests, optimization of dosage and pH, regeneration experiments, and applications on real industrial wastewater to validate its potential for large-scale implementation.

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