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Research Article

Kinetics and Adsorption Equilibrium of Methylene Blue Using Shell and Coconut Husk Ash from Waste of Tofu Industry Cooking Process Activated with Sulfuric Acid

Eny Yulianti [1]*, Falahiyah [1], Fariz Rifqi Zul Fahmi[2], Vina Nurul. Istighfarini [1], Nur Aini [1]

[1]Chemistry, Faculty of Science and Technology, UIN Maulana Malik Ibrahim, Malang, 65144, Indonesia
[2]Mechanical Engineering, Faculty of Science and Technology, UIN Maulana Malik Ibrahim, Malang, 65144, Indonesia

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*Email Corresponding:
enyyulianti@kim.uin-malang.ac.id

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Methylene blue adsorption was conducted using ash from coconut husks and shells, derived from industrial tofu fuel waste in Malang City. The ash was activated using 2% H_2SO_4. The determination of the optimum adsorption conditions for methylene blue included variations in contact time, solution pH, and adsorbent dosage. This study also examined adsorption capacity and adsorption isotherms. The ash was characterized using XRD to identify the silica phase present in the ash. The results showed that the optimum conditions for adsorbing 50 mL of methylene blue at a concentration of 13.97 mg/L were a contact time of 75 minutes, a pH of 3, and an adsorbent dosage of 0.5 grams. The adsorption capacity of methylene blue was found to be 4.533 mg/g, and the adsorption isotherm of methylene blue using H_2SO_4-activated coconut husk and shell ash followed the Freundlich isotherm model more closely than the Langmuir isotherm. XRD analysis indicated that the crystallinity and purity of silica in the ash activated with 2% H_2SO_4 improved due to the removal of impurity peaks from KCl and $NaHCO_3$.

Keywords: Coir Ash, Coconut Shell ash, Methylene blue, Adsorption isotherm, Sulfuric acid

1. Introduction

The dye waste produced by industries is generally composed of non-biodegradable organic compounds, which cause environmental pollution, particularly in aquatic ecosystems [1]. One of the most commonly used dyes is methylene blue (MB). In the dye industry, it is used as a paper dye in combination with other dyes, while in laboratories, it serves as an indicator dye. Methylene blue contains benzene groups, making it difficult to degrade [2]. This compound is toxic, can cause genetic mutations, and affects reproduction.

An effective method for treating dye wastewater is the adsorption process. Adsorption occurs due to the attractive forces between adsorbate molecules and active sites on the surface of the adsorbent [3]. According to [4], one of the best types of adsorbents for reducing dye concentration in solutions is silica-containing material derived from combustion waste (ash). M. Kumalasari et al. [5] stated that ash has high effectiveness as an adsorbent due to its silica content, which reaches 80-90%. The study of Baunsele et al. [6] found that the optimum adsorption of methylene blue occurred at pH 7 over a duration of 75 minutes, with an adsorption capacity of 1.41 mg/g.

The adsorption capacity of an adsorbent can be enhanced through chemical or physical activation processes [7]. Tang [8] stated that activation with the addition of sulfuric acid increases the adsorption capacity of methylene blue by 7.99 mg/g. Kumar et al. [9] activated fly ash with H_2SO_4, which enhanced the removal of RR194 dye, reducing its concentration by up to 96.6%. One such investigation explored Fe adsorption was performed using 150-180 mesh fly ash powder activated with concentrated H_2SO_4, achieving a 94% adsorption efficiency[10].

Solid waste from the tofu industry, particularly ash derived from the combustion of coconut shells and husks, is generally disposed of without further utilization and may contribute to environmental pollution. However, this ash contains silica and other minerals with great potential to be used as an adsorbent material. Utilizing this waste not only reduces the volume of solid waste generated by the tofu industry but also adds value through the development of a low-cost and environmentally friendly adsorbent for removing dyes such as methylene blue. Therefore, the use of coconut shell and husk ash as an adsorbent supports the concept of waste-to-resource while providing a practical solution for managing tofu industry waste.

In this study, the chemical activation of coconut husk and shell ash was carried out using 2% H_2SO_4, and the optimal conditions for the methylene blue adsorption process were determined. The ash was characterized using XRD to identify the mineral phases present.. Additionally, the adsorption isotherm model will be studied by comparing the Langmuir and Freundlich models.

2. Materials and Methods

2.1 *Materials*

The materials used include ash from coconut husks and shells obtained from the tofu industry in Malang City, H_2SO_4 (Merck 96%), distilled water, and methylene blue (Merck p.a.).

2.2 Methods

2.2.1 Ash Preparation

The preparation of coconut husk and shell ash begins with washing them using distilled water, followed by drying at 110°C for 3 hours. The resulting solid is then ground using a mortar and sieved with a 120-mesh sieve.

2.2.2 Ash Activation

The ash activation process involves adding 100 mL of 2% H_2SO_4 solution to 100 grams of ash in a beaker. The ash is soaked for 24 hours and then filtered using filter paper. The obtained residue is dried in an oven at 60°C for 24 hours. The residue is then washed until the filtrate reaches a pH of 7. After that, the ash is dried at 105°C for 24 hours. The activated ash is then characterized using XRD.

2.2.3 Standard Curve Determination

The adsorption of methylene blue begins with determining the optimal wavelength for a 5 PPM methylene blue solution. The optimal wavelength is then measured using a UV-Vis spectrophotometer within the 600-700 nm range. Next, the stability time is determined at intervals of 0, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, and 130 minutes at the optimal wavelength. A graph is then created to illustrate the relationship between absorbance and operational time. Subsequently, a calibration curve for methylene blue is prepared using concentrations of 0, 1, 2, 3, 4, and 5 PPM, with absorbance measured at the optimal wavelength. A standard curve is then plotted to represent the relationship between concentration and absorbance, yielding the equation of the line $y = ax + b$[11].

2.2.4 Determination of Optimal Conditions

The determination of the optimal conditions for methylene blue adsorption was carried out with several variations, including contact time. A total of 50 mL of a 10 PPM methylene blue solution was added to an Erlenmeyer flask containing 0.5 grams of ash, then covered with aluminum foil. The sample was shaken at 100 rpm with varying contact times of 15, 30, 45, 60, 75, and 90 minutes. The adsorption was analyzed using UV-Vis spectroscopy at the optimal wavelength.

The pH variation was conducted by preparing 50 mL of a 10 PPM methylene blue solution with pH values of 3, 5, 7, 9, and 11, which was then added to an Erlenmeyer flask containing 0.5 grams of ash. The sample was shaken at 100 rpm for the optimal contact time. After filtration, the remaining methylene blue concentration and absorbance were measured. For the adsorbent dosage variation, 50 mL of a 10 PPM methylene blue solution at its optimal pH was added to separate Erlenmeyer flasks containing 0.5, 1.0, 1.5, 2.0, and 2.5 grams of ash. The flasks were then covered with aluminum foil, and the samples were shaken at 100 rpm for the optimal contact time. After filtration, the remaining methylene blue concentration and absorbance were measured.

2.2.5 Determination of Adsorption Isotherm and Adsorption Capacity

A total of 50 mL of methylene blue solution was prepared for each concentration variation of 10, 20, 30, 40, and 50 PPM at the optimal pH[11]. The solutions were then added to Erlenmeyer flasks containing ash at the optimal dosage. The samples were covered with aluminum foil, shaken at 100 rpm for the optimal contact time, filtered, and their concentration and absorbance were measured. The adsorption capacity and adsorption percentage can be calculated using the following equations:

$$\%\ \text{Adsorbed} = \frac{Co-Ce}{Co}\ x\ 100\% \quad (1)$$

$$Qv = \frac{(Cu-Ce)V}{W} \quad (2)$$

$$\frac{Ce}{Qe} = \frac{1}{(Qo\ .b)} + \frac{Ce}{Qo} \quad (3)$$

$$\ln \mathrm{Qe} = \ln \mathrm{Kf} + \frac{1}{n} \ln \mathrm{Ce} \quad (4)$$

The adsorption capacity (Qe, mg/g) was calculated based on the initial concentration (Co, mg/L), the equilibrium concentration (Ce, mg/L), the solution volume (V, L), and the adsorbent weight (W, g). The Langmuir constants were represented by Qo and b, while the Freundlich constants were represented by Kf and n, which indicate the adsorption capacity and adsorption intensity, respectively.

The adsorption process was evaluated under optimal conditions for methylene blue adsorption using ash, following two isotherm models: the Langmuir model and the Freundlich model[12]. The Langmuir isotherm equation was determined by plotting the relationship between Ce and Ce/Qe, resulting in an R^2 value from the linear equation y = ax + b, which was then converted using Equation 3. Meanwhile, the Freundlich isotherm equation was determined by plotting the relationship between ln Ce and ln Qe, yielding a linear equation and regression value, which was then converted using Equation 4.

3. Result and Discussion

3.1 *Preparation and Activation of Coconut Husk and Shell Ash with 2% H_2SO_4*

The ash was prepared by washing it with distilled water to remove impurities attached to the coconut husk and shell ash. It was then dried at 110°C for 3 hours to evaporate the moisture within the ash pores. Once dried, the ash was ground and sieved using a 120-mesh sieve to ensure a uniform particle size. If the ash particles are not uniform, the adsorption contact area on the ash surface will vary, leading to less optimal adsorption performance[13]. Smaller particle sizes provide a larger surface area, which increases the adsorption rate.

The activation treatment using H_2SO_4 solution helps dissolve impurities in the ash material, allowing the pore openings to become more exposed, which in turn increases the specific surface area of the pores. Additionally, the number of active sites also increases as previously hidden sites become accessible, and new active sites may emerge due to the dissolution reaction that replaces the cations in the coconut husk and shell ash with H^+ ions from the acid solution. According to trung et al study [14], the activation of ash using sulfuric acid facilitates ion exchange, replacing the cations in the ash structure with H^+ ions, which belong to the Bronsted acid group.

3.2 *Characterization of Activated Ash using XRD*

Coconut husk and shell ash have the ability to act as an adsorbent due to their silica content. This is demonstrated by the XRD analysis results of both non-activated and H_2SO_4 2%-activated ash, as presented in **Figure 1**.

The XRD analysis results indicate that non-activated ash contains characteristic peaks corresponding to $HNaCO_3$ (Nahkolite) at 42%, KCl (Sylvite) at 19%, and SiO_2 (Coesite, Stishovite) at 39%. In contrast, the H_2SO_4 2%-activated ash exhibits only characteristic silica peaks with 100% purity. This suggests that the activation process using H_2SO_4 effectively enhances silica purity by eliminating impurities such as KCl and $HNaCO_3$, thereby improving the ash's adsorption capacity for methylene blue.

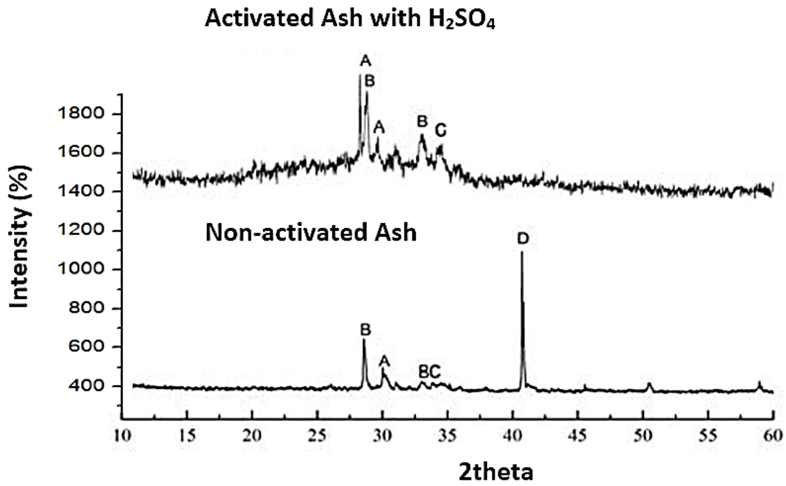

Figure 1. The XRD analysis results of coconut husk and shell ash indicate the presence of various compounds: A = SiO_2 (Silica), B = SiO_2 (Coesite), C = SiO_2 (Stishovite), and D = KCl (Sylvite)

3.3 Determination of Optimum Adsorption Conditions for Methylene Blue

3.3.1 Determination of Methylene Blue Wavelength

In this study, the determination of the optimum wavelength for methylene blue was conducted within the range of 600-700 nm using a UV-Vis spectrophotometer. The optimum wavelength is the wavelength that exhibits the highest sensitivity, a flat absorbance curve, and ensures compliance with Lambert-Beer's law. Additionally, under these conditions, measurement errors due to wavelength recalibration are minimized [15]. In this study, the optimum wavelength for methylene blue was found to be 664.9 nm with an absorbance of 0.917.

3.3.2 Determination of Methylene Blue Stability Time

The determination of stability time aims to identify the optimal and stable measurement time for methylene blue. Stability time is determined by analyzing the relationship between measurement time and the absorbance of the methylene blue solution. According to Gandjar [15], at the beginning of the reaction, the absorbance of this colored compound increases until it reaches a certain point where it stabilizes. However, with prolonged measurement time, the colored compound may degrade or decompose, leading to a decrease in color intensity and, consequently, a drop in absorbance. For this reason, the measurement of colored compounds should be conducted during their stability period. In this study, methylene blue was found to have a stability time range between 10 to 110 minutes. The curve representing the relationship between stability time and absorbance is shown in **Figure 2**.

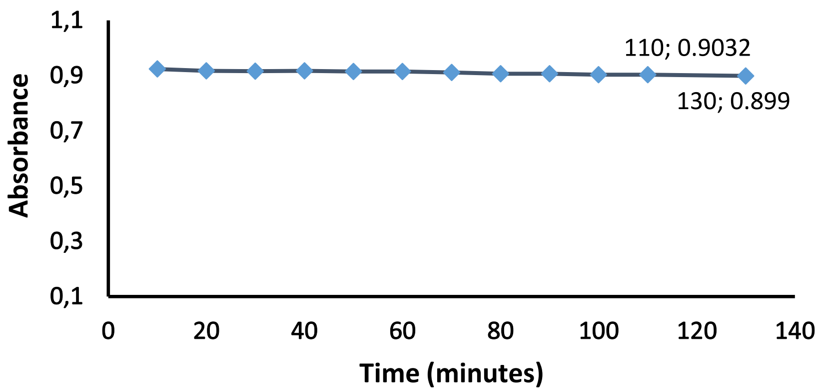

Figure 2. Curve of the Relationship Between Stability Time and Absorbance

3.3.3 *Preparation of the Methylene Blue Calibration Curve*

The standard curve for the methylene blue solution resulted in a linear regression equation: $y = 0.224x – 0.0136$, with $R^2 = 0.9971$, where y represents absorbance and x represents the concentration of methylene blue. Since the regression coefficient (R^2) is very close to 1, the relationship between absorbance and concentration is highly linear and aligns with Lambert-Beer's Law.

3.3.4 *Optimum Contact Time*

Contact time refers to the duration required for the adsorption process to take place between coconut husk and shell ash as the adsorbent and methylene blue as the adsorbate. A longer contact time allows for better diffusion and attachment of adsorbate molecules onto the adsorbent surface. In this study, variations in contact time were examined to determine the optimum contact time, as it significantly influences adsorption equilibrium. Contact time affects the attractive forces or interactions between the adsorbent and adsorbate, such as Van der Waals forces and electrostatic interactions. The results of the contact time variations are presented in **Figure 3**.

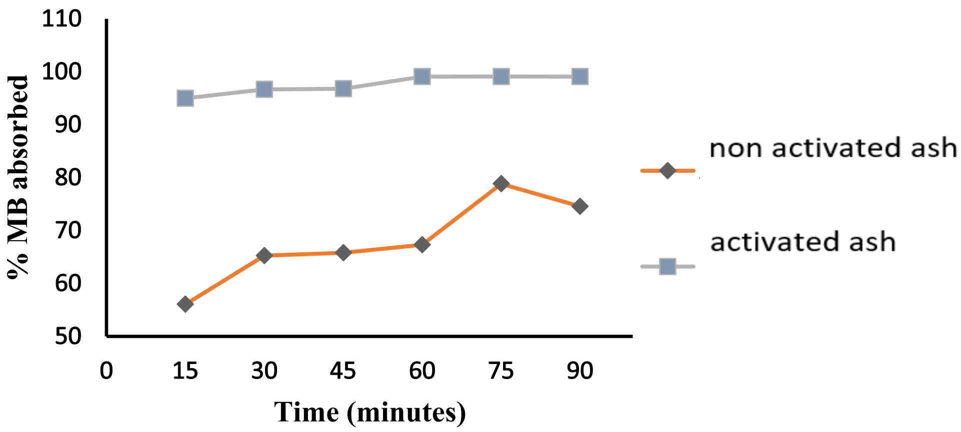

Figure 3. Curve of the Relationship Between Contact Time and % Methylene Blue Adsorbed

Figure 3 shows that the optimum contact time for methylene blue adsorption using non-activated ash and H_2SO_4-activated ash is 75 minutes, with methylene blue adsorption percentages of 78.81% and 99.1%, respectively.

3.3.5 Optimum pH

In the adsorption process, the ionization of the adsorbate depends on the pH conditions. The pH variation in this study aimed to determine the optimum pH and the adsorption pattern of methylene blue, whether it occurs through physical or chemical interactions. The results showed that the optimum pH for adsorption using H_2SO_4-activated ash was pH 3, with 99.1% methylene blue adsorbed, while for non-activated ash, the optimum pH was pH 5, with 79.82% methylene blue adsorbed. The curve illustrating the relationship between pH and the percentage of methylene blue adsorbed is presented in **Figure 4**.

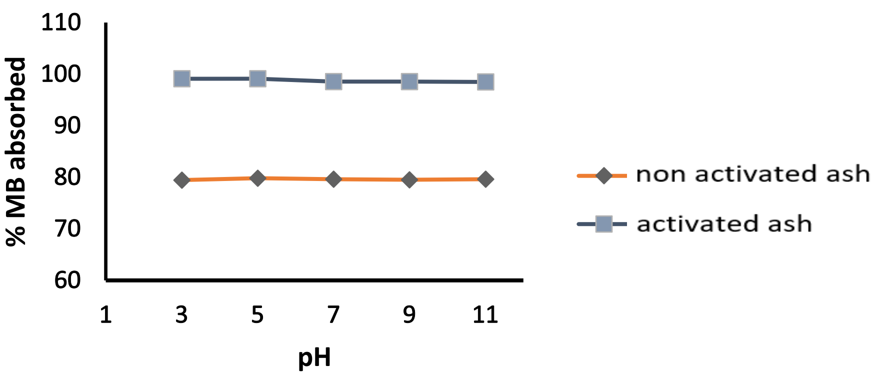

Figure 4. Curve of the relationship between pH and the percentage of methylene blue absorbed.

Figure 4 shows that methylene blue adsorption is not influenced by pH conditions, suggesting that the adsorption process occurs physically rather than chemically.

3.3.6 *Optimum Adsorbent Dosage*

Adsorbent dosage is one of the key factors influencing the adsorption process. The higher the adsorbent dosage used, the more effective the adsorption process becomes. In this study, variations in adsorbent dosage were tested to determine the optimum dosage for methylene blue adsorption. The results of the adsorbent dosage variations are presented in **Figure 5**.

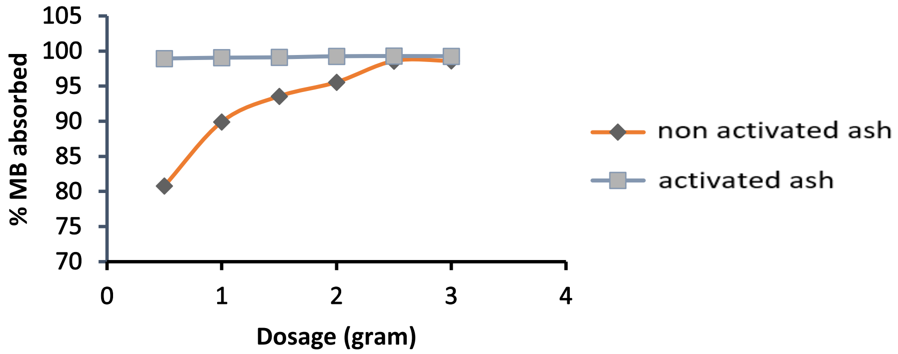

Figure 5. Curve Representing the Relationship Between Adsorbent Dosage and Percentage of Methylene Blue Adsorbed

Figure 5 shows that the optimum adsorbent dosage for methylene blue adsorption using H_2SO_4-activated ash is 0.5 g, with 98.94% of methylene blue adsorbed, whereas for non-activated ash, the optimum dosage is 2.5 g, with 98.59% adsorption. This indicates that activated ash has better adsorption capability than non-activated ash. The activation process with 2% H_2SO_4 increases the purity of active sites by removing impurities that previously covered them, thereby enhancing adsorption efficiency.

3.3.7 Determination of Adsorption Capacity and Adsorption Isotherm

Adsorption capacity refers to the amount of adsorbate mass absorbed per unit mass of adsorbent. The determination of adsorption capacity was carried out by varying the concentration of methylene blue, and the obtained data were analyzed using Equation 2. The curve representing the relationship between methylene blue concentration and the percentage of methylene blue adsorbed is presented in **Figure 6**.

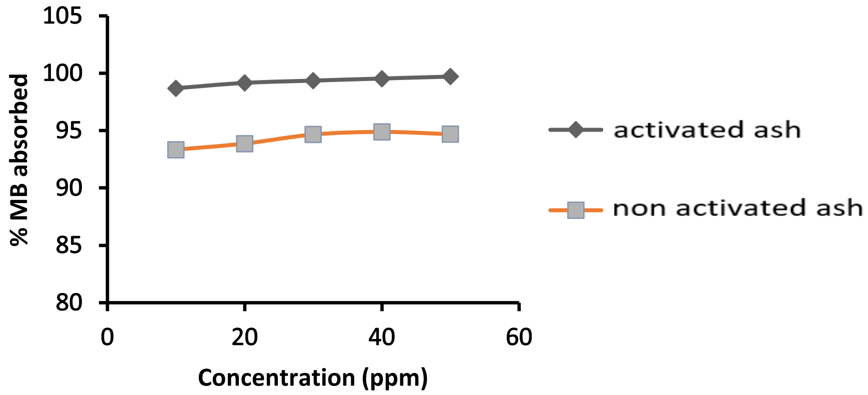

Figure 6. The curve showing the relationship between concentration and the percentage of methylene blue absorbed

Figure 6 shows that sulfuric acid-activated ash exhibits a higher adsorption capacity for methylene blue compared to non-activated ash. The activated ash was able to adsorb 99.81% of 50 mL methylene blue at a concentration of 50 PPM, with an adsorption capacity of 4.533 mg/g. In contrast, the non-activated ash achieved a maximum adsorption percentage of 94.89% at 50 PPM, with an adsorption capacity of 0.86 mg/g.

The adsorption process of methylene blue dye can be analyzed by testing the linear adsorption isotherm equation at equilibrium conditions. In this study, the adsorption isotherm approach was conducted using the Langmuir and Freundlich isotherm models.

3.3.8 *Langmuir Isotherm*

The Langmuir adsorption isotherm assumes that the adsorbent has a homogeneous surface. Each adsorbent molecule can only adsorb a single adsorbate molecule, forming a monolayer. This Langmuir isotherm theory also applies to chemical adsorption, where a monolayer is formed [16]. The Langmuir isotherm is determined by following Equation 3.

The determination of the Langmuir adsorption isotherm equation can be obtained by relating the values of Ce and Ce/Qe. Adsorption follows the Langmuir isotherm if the regression coefficient (R^2) value approaches 1. The value of 1/Qo represents the slope of the line, indicating the adsorption capacity of methylene blue (mg/g), while b (mg/L) is the Langmuir constant that relates adsorption capacity to adsorption energy. The values of R^2, Qo, and b in the Langmuir equation are presented in **Table 1**. Langmuir Isotherm Graph depicted in **Figure 7** and **8**.

Table 1. Langmuir Isotherm Value

Ash	Langmuir Adsorption Isotherm		
	Q_o (mg/g)	b (L/mg)	R^2
Non-activated	-2,209	-0,122	0,6652
Activated with H_2SO_4	0,470	-12,126	0,7526

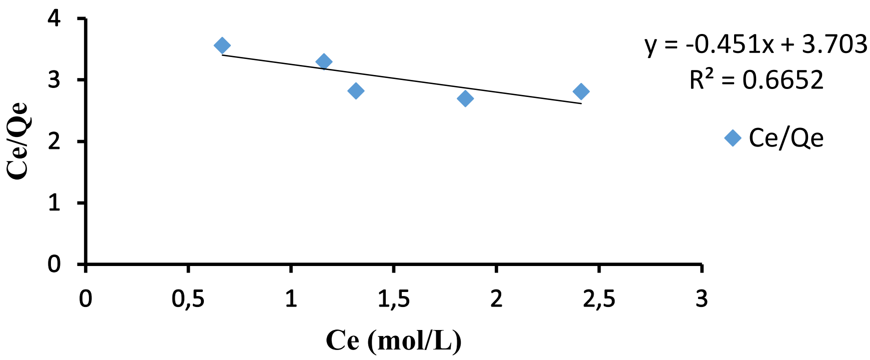

Figure 7. Langmuir isotherm curve of non-activated ash

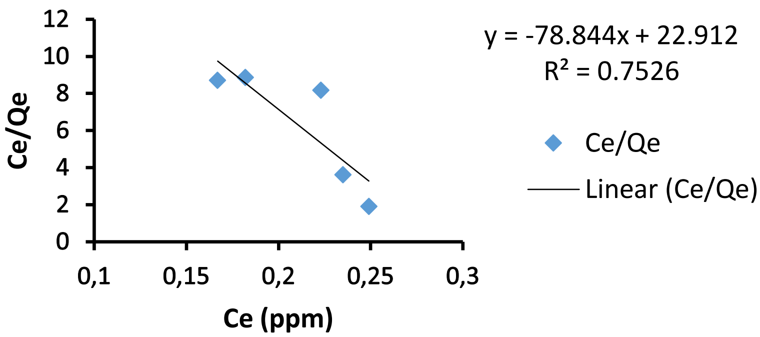

Figure 8. Langmuir adsorption isotherm of methylene blue on H_2SO_4-activated ash

3.3.9 *Freundlich Isotherm*

The assumption of the Freundlich adsorption isotherm is that the adsorbent has a heterogeneous surface. Each adsorbent molecule has a different adsorption potential (multilayer). The Freundlich isotherm assumption applies to physical adsorption, forming a multilayer [17]. The Freundlich adsorption isotherm can be determined by plotting In Ce against In Qe. The linear equation for the Freundlich isotherm follows Equation 5. The values for the Freundlich adsorption isotherm are presented in **Table 2**. Freundlich Isotherm Curve illustrated in **Figure 9** and **10**.

Table 2. Freundlich Isotherm Value

Ash	Freundlich Adsorption Isotherm			
	K_f (mg/g)	n	1/n	R^2
Non-activated	0,3111	0,823	-1,22	0,9912
Activated with H_2SO_4	0,0018	-0,30	-3,23	0,9837

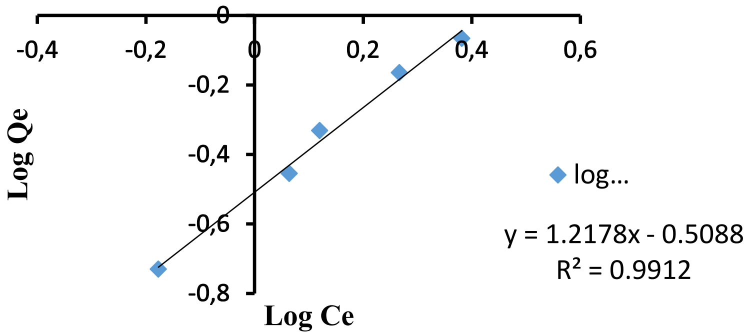

Figure 9. Freundlich isotherm curve of non-activated ash

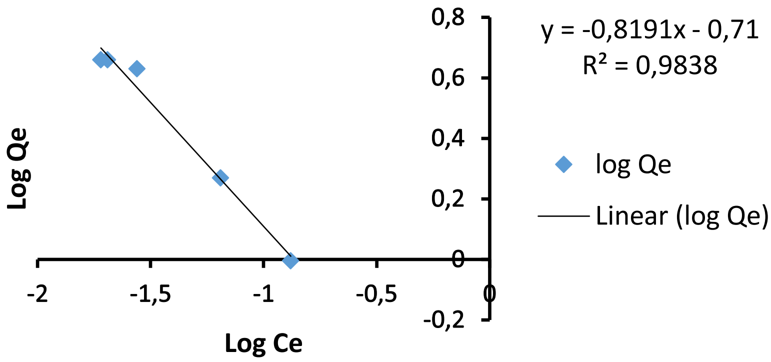

Figure 10. Freundlich adsorption isotherm of methylene blue on H_2SO_4-activated ash

Based on the comparison of the Langmuir and Freundlich isotherm results in **Table 1** and **Table 2**, the adsorption of methylene blue using sulfuric acid-activated coconut husk and shell ash tends to follow the Freundlich isotherm, as indicated by the R^2 value of 0.9837. Similarly, the adsorption using non-activated ash also follows the Freundlich isotherm, with an R^2 value of 0.9912.

Additionally, based on the comparison of the 1/n values, where n represents the adsorption intensity, [18] stated that if the 1/n value approaches 0, the adsorbent surface is heterogeneous. If 1/n < 1, the adsorption follows the Langmuir isotherm, while if 1/n > 1, the adsorption is cooperative. In this study, the adsorption of methylene blue tends to follow the Freundlich isotherm, as the 1/n values are close to 0, specifically -3.23 and -1.22. This indicates that the adsorbent surface is predominantly heterogeneous and that the adsorption process occurs physically.

4. Conclusion

The optimal adsorption conditions for methylene blue using acid-activated coconut husk and shell ash (H_2SO_4 2%) were achieved at a contact time of 75 minutes, pH 3, and an adsorbent dose of 0.5 grams in 50 mL of 13.97 PPM methylene blue solution. The adsorption capacity was 4.533 mg/g in 50 mL of 45.417 mg/L methylene blue solution, with the adsorption process following the Freundlich isotherm model. XRD analysis of the H_2SO_4-activated coconut husk and shell ash indicated an increase in the purity and crystallinity of silica, as evidenced by the disappearance of impurity peaks for KCl and $HNaCO_3$.

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