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Study on the Effectiveness of H_2SO_4 Activated Coffee Grounds and Imperata cylindrica Bioadsorbents for Methylene Blue Adsorption

Unung Leoanggraini[1], Tifa Paramitha[1], Rahma Ardelia Raissa[1], Mutiara Nur Annisa[1], Muhammad Salman Alfarisi[1], Radianti Novita Sari[1] , Rony Pasonang Sihombing[1*]

[1]Department of Chemical Engineering, Politeknik Negeri Bandung, Indonesia

[*]Corresponding Author: rony.pasonang.sihombing@polban.ac.id

Abstract
Textile wastewater with methylene blue dye causes serious environmental issues because of its toxicity, persistence, and negative effects on aquatic life. Using agricultural waste as bioadsorbents provides a sustainable and low-cost way to remove dye from wastewater. The goal of this study was to assess how effective H_2SO_4-activated bioadsorbents made from coffee grounds and Imperata cylindrica are for methylene blue adsorption in batch systems. We prepared the bioadsorbents by carbonizing them at 450°C, followed by chemical activation with 2 M sulfuric acid. We characterized them using FTIR and SEM to study functional groups and surface shape. We conducted batch adsorption experiments by changing the adsorbent mass (0.15–0.3 g), contact time (30–120 minutes), and methylene blue concentration (10–100 mg/L). Langmuir and Freundlich isotherm models were used to evaluate the adsorption behavior. The results showed that activated coffee grounds and Imperata cylindrica had improved porosity and changes in functional groups after activation. The best adsorption conditions were found with an adsorbent mass of 0.3 g, a contact time of 120 minutes, and a methylene blue concentration of 10 mg/L, reaching an adsorption efficiency of up to 97.498%. The adsorption process fit the Langmuir isotherm model better, with maximum adsorption capacities of 42.735 mg/g for coffee grounds and 13.210 mg/g for Imperata cylindrica. These findings suggest that coffee grounds and Imperata cylindrica are promising, cost-effective bioadsorbents for removing methylene blue in textile wastewater treatment.

Keywords: Bioadsorbent, Coffee grounds, Imperata cylindrica, Methylene blue, Batch adsorption.

INTRODUCTION
The increasing amount of dye-containing wastewater generated by the textile industry is directly related to the environmental impacts of the dyeing process. Textile wastewater often contains dye compounds at concentrations of approximately 20–30 mg/L, which are difficult to degrade naturally (Kustiningsih et al., 2017). According to the Regulation of the Minister of Environment and Forestry of the Republic of Indonesia No. 6 of 2021, the maximum allowable concentration of methylene blue (MB) in wastewater discharge is 5 mg/L. MB is a synthetic cationic heterocyclic aromatic dye widely used in the textile, medical, pharmaceutical, and analytical chemistry industries (Annisah et al., 2025; Dwijayanti et al., 2020; Ginting et al., 2019; Irnameria, 2020). It has a molecular weight of 319.86 g/mol, a melting point of 105°C, and high water solubility of 4.36×10^4 mg/L (Dwijayanti et al., 2020; Ginting et al., 2019; Indah Fajarwati et al., 2016; Musfirah Adhar et al., 2022). Its stable aromatic structure makes MB persistent in the environment, while its release into aquatic systems can reduce light penetration, inhibit photosynthesis, and disrupt aquatic ecosystems (Hesty et al., 2023; Nuur Hanifah et al., 2023). Approximately 10–15% of synthetic dyes used in industrial processes are estimated to be discharged into wastewater, contributing significantly to water pollution and ecological damage (Irfandy et al., 2021; Irvianti, 2019). Therefore, the persistence and potential toxicity of MB necessitate the development of effective, low-cost, and sustainable wastewater treatment technologies that are adaptable to local environmental regulations.

Among various wastewater treatment technologies, adsorption is widely regarded as a promising method for dye removal because of its efficiency, simplicity, reusability, and economic feasibility (Lucas Hendrajaya et al., 2024; Pengajar et al., 2017). Activated carbon is one of the most commonly used adsorbents due to its high surface area, porosity, and abundant surface functional groups, which facilitate the adsorption of pollutants. Activated carbon is generally produced from carbon-rich materials through carbonization and activation processes to enhance its porosity and adsorption capacity. However, the relatively high cost of commercial activated carbon has encouraged the exploration of alternative precursors derived from abundant and renewable agricultural wastes. Various agricultural by-products, including coconut husk, rice husk, coffee grounds, and *Imperata cylindrica*, have been investigated as potential raw materials for activated carbon and bioadsorbents.

Coffee grounds are an abundant agricultural by-product with considerable potential as an adsorbent precursor. They contain approximately 47.8–58.9% carbon, 21% cellulose, 1.9–2.3% nitrogen, and 0.43–1.6% ash, providing a carbon-rich structure suitable for conversion into activated carbon (Nurhidayanti et al., 2021; *Efektivitas Adsorben Berbasis Alginat dan Karbon Aktif Ampas Kopi dalam Menurunkan Kadar COD Limbah Cair Industri Tahu*, n.d.). Previous studies have demonstrated the potential of coffee-ground-derived activated carbon for MB removal, with adsorption performance reported at MB concentrations of up to 45.93 mg/L under batch conditions (Febrianti et al., 2023; Hidayah et al., 2022; Maflihah et al., 2022). These findings indicate that coffee grounds can serve as a low-cost and sustainable carbon precursor for dye removal.

In addition to coffee grounds, *Imperata cylindrica*, commonly known as reed grass, is another abundant biomass resource with potential as a bioadsorbent. It contains approximately 5.42% ash, 3.6% silica, 18.12% lignin, and 40.28% alpha-cellulose (Balqis Wiyana et al., 2022; Yudistirani et al., 2022). Its high cellulose content provides oxygen-containing functional groups, such as hydroxyl and carboxyl groups, which can interact with dye molecules through various adsorption mechanisms (Adsorpsi et al., 2022; Siagian, 2020). Previous research using *I. cylindrica* ash reported an adsorption capacity of 0.4844 mg/g, demonstrating its potential for pollutant removal (Asiah et al., 2022; Badriyah & Putri, 2017; Huda & Yulitaningtyas, 2018). Nevertheless, most previous studies have investigated coffee grounds and *I. cylindrica* individually, while their combined use as a composite bioadsorbent remains relatively underexplored.

Coffee grounds and *I. cylindrica* were selected because they are readily available and low-cost biomass resources with complementary physicochemical characteristics. Coffee grounds have a relatively high carbon content of approximately 47.8–58.9%, making them suitable precursors for porous activated carbon, whereas *I. cylindrica* contains approximately 40.28% alpha-cellulose and 18.12% lignin, which may contribute oxygen-containing functional groups involved in methylene blue adsorption. Combining these materials was therefore intended to integrate the carbon-rich porous structure of coffee-ground-derived carbon with the lignocellulosic surface functionalities of *I. cylindrica*-derived carbon. This combination potentially provides more diverse adsorption sites than either precursor alone. However, because the present study did not directly compare the mixed bioadsorbent with each individual adsorbent under identical operating conditions, synergistic superiority is not claimed. Chemical activation using sulfuric acid (H_2SO_4) can further modify the biomass structure and enhance the availability of adsorption sites. However, the adsorption performance of a sulfuric-acid-activated composite derived from coffee grounds and *I. cylindrica* for MB removal in batch systems has not been sufficiently investigated. Therefore, this study aims to synthesize a composite bioadsorbent from coffee grounds and *I. cylindrica* activated with H_2SO_4 and to evaluate its adsorption performance and optimal operating conditions for MB removal in a batch system. The contribution of this study lies in evaluating an H_2SO_4-activated mixed bioadsorbent prepared from coffee grounds and *I. cylindrica* for methylene blue removal and in determining its best adsorption conditions within the investigated experimental ranges.

METHODS

Materials and Equipment

The materials used for this series of studies were robusta coffee grounds, reed grass, methylene blue, distilled water, 2M sulfuric acid (H_2SO_4), and 0,1 M hydrochloric acid (HCl). The equipment used for this series of studies includes a furnace, oven, chopper, sieve shaker, UV-Vis spectrophotometer, hot plate, analytical balance, pH indicator paper, desiccator, filter paper, chemical glassware, Erlenmeyer flasks, and measuring flasks. The main instruments used were a UV-Vis spectrophotometer, Fourier Transform Infrared Spectroscopy (FTIR), and a Scanning Electron Microscope-energy (SEM).

Adsorbent Preparation

The reed grass was taken apart from its stems and cleaned with water. Meanwhile, the coffee grounds were made ready and left in hot water for 10 minutes. After that, both materials were placed in an oven and heated at 100 degrees Celsius for three hours. Then, they were passed through a sieve with 60 mesh, which is about 250 micrometers in size. The dry coffee grounds and reed grass were each burned in a furnace at 450 degrees Celsius for 25 minutes to turn them into carbon. The carbon was treated with a solution of 2 M sulfuric acid by soaking it for 24 hours in 150 mL of the solution. Once the carbon was turned on, it was passed through a filter, then rinsed with clean water until it had a neutral pH, and finally dried in an oven set to 100°C for four hours.

Batch Adsorption Procedure

The experiments were conducted at ambient laboratory temperature (25–30 °C). The initial pH of the methylene blue solution was 7, and no pH adjustment was performed during the adsorption experiments.

Adsorbent Characterization (SEM and FTIR)

The characteristics of activated carbon from coffee grounds and reed grass were tested using FTIR (Fourier Transform Infrared) to determine the functional groups contained in the activated carbon samples before and after the adsorption process. In addition, the characteristics of the activated carbon samples were also tested using SEM (Scanning Electron Microscope-energy) to determine the surface morphology of the adsorbent.

Concentration Analysis and Calculation

The adsorption process involves the accumulation of substances from a fluid phase onto the surface of a solid material, forming a thin layer of adsorbate. It is recognized for its simplicity, reusability, and cost-effectiveness (Lucas Hendrajaya et al., 2024; Pengajar et al., 2017). In this study, the adsorption performance of a composite bioadsorbent consisting of coffee grounds and *Imperata cylindrica*, chemically activated with sulfuric acid, was evaluated for the removal of methylene blue (MB) in a batch system under various operating conditions. Following the batch adsorption experiments, the adsorption test solutions were analyzed using a UV–Vis spectrophotometer at a wavelength range of 500–700 nm to determine the MB concentration before and after the adsorption process. The adsorption capacity (Q) was calculated using the following equation:

$$Q = \left(\frac{(C_0 - C)\,V}{W}\right) \times 100\% \qquad (1)$$

where Q is the adsorption capacity (mg/g), C_0 is the initial concentration of MB (mg/L), C is the MB concentration at time t (mg/L), V is the solution volume (L), and W is the adsorbent mass (g) (Fachria et al., 2019; Sy et al., 2016).

RESULTS AND DISCUSSION

Preparation of Coffee Grounds and Reed Grass Activated Carbon

From the results of the carbonization process, the yield of activated carbon from coffee grounds and reed grass was 60% and 20,15%, respectively. Then, chemical activation was carried out by soaking in 2M H_2SO_4 activator solution for 24 hours. Then neutralization was carried out using distilled water and dried in the oven.

Characterization of Bioadsorbents (Moisture Content, Ash Content, Volatille Matter, and Fixed Carbon)

The activated carbon of coffee grounds and reed grass was then tested to ensure that the activated carbon produced met the technical activated carbon standard referring to SNI 06-3730-1995. The results obtained from the characterization test are as follows.

Table 1. Characterization Test of Coffee Grounds and Reed Grass

Parameters	SNI 06-3730-1995	Types of activated carbon	
		Coffee Grounds	Reed Grass
Moisture content	Maximum 15%	4,28%	6,30%
ash content	Maximum 10%	0,95%	6,18%
volatile matter	Maximum 25%	3,1%	50,24%
fixed carbon	Minimum 65%	95,97%	43,46%

Based on the test results, it was found that the activated carbon of coffee grounds and reed grass produced met the SNI 06-3730-1995 standard. As for the volatile matter test, the reeds amounted to 50.24% of the maximum provision of 25%. This is due to the influence of carbonization temperature, the higher the temperature, the more organic compounds are lost so that the content of volatile substances in activated carbon is getting smaller (Fachria et al., 2019; Sy et al., 2016). In addition, the fixed value of reed grass carbon produced still does not meet the minimum standard of 65%.

FTIR Characterization

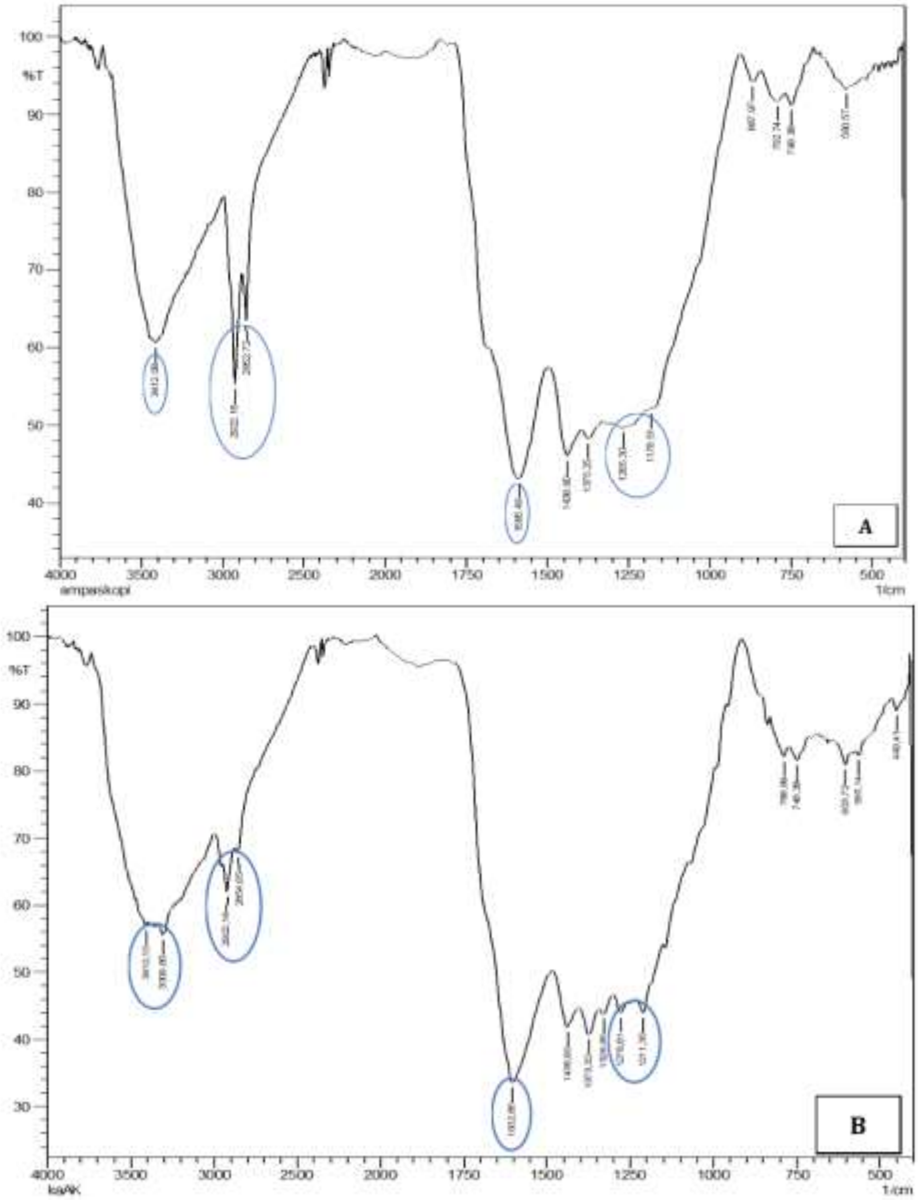


Figure 1. (A) FTIR spectrum of coffee grounds carbon before activation (B) FTIR spectrum of coffee grounds carbon after activation

The results of FTIR measurements of coffee grounds before and after activation obtained peaks that appear indicate the presence of several functional groups.

Table 2. FTIR spectrum of coffee grounds Carbon before and after activation

Peak	Coffee grounds carbon before activation (cm^{-1})	Coffee grounds carbon after activation (cm^{-1})	Wavelength (cm^{-1})	Functional groups
1	3412,08	34101,5	3200-3600	O-H
2	2852,72	2922,16	2340-3000	C-H
3	1585,49	16028,5	1500-1600	C=C
4	1265,30	12788,51	1000-1300	C-O

From the above table, it can be observed that after activation, there is a decrease in transmittance in the peak corresponding to C=C. This indicates an increase in aromatic carbon. In addition, a decrease in peak transmittance also occurs in the C-O oxygen group indicating that activation reduces the amount of oxygen component.

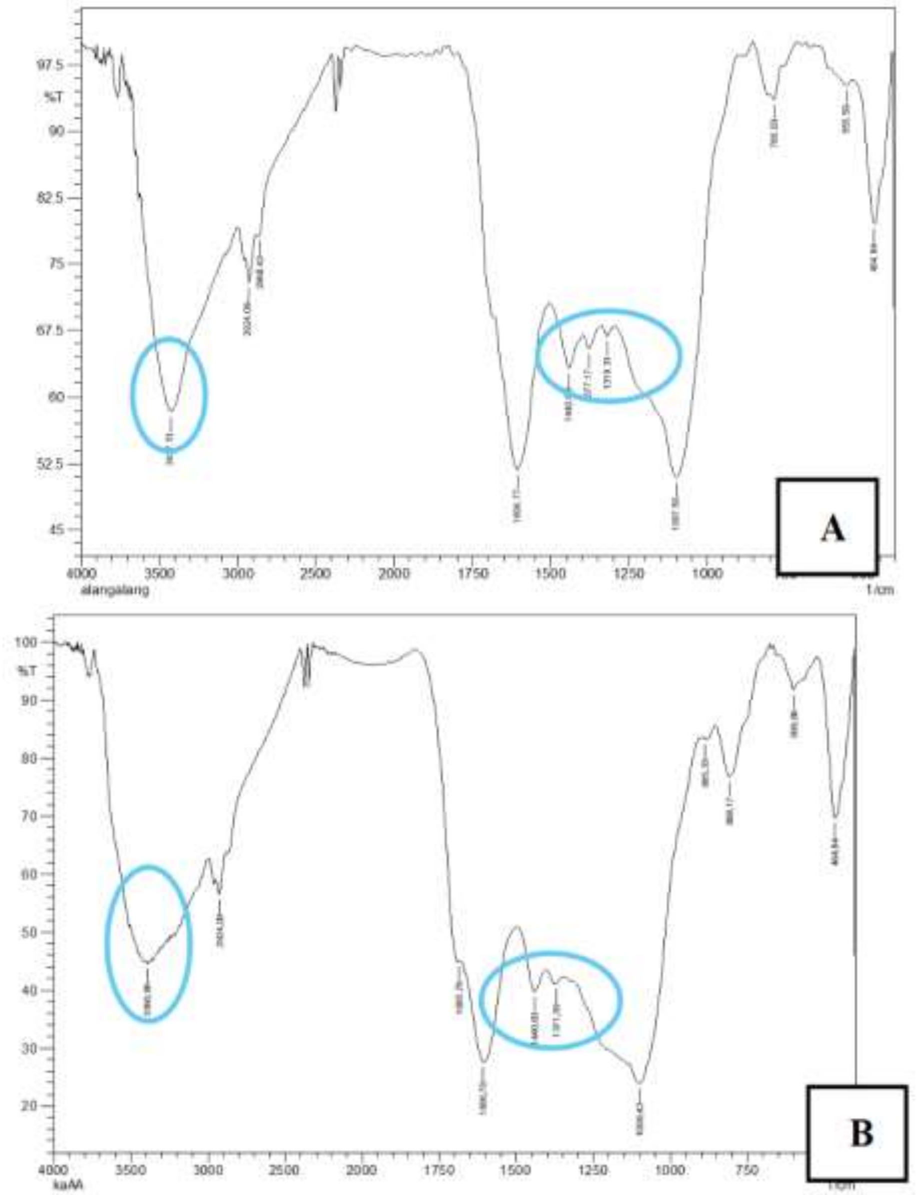


Figure 2. (A) FTIR spectrum of reed grass carbon before activation (B) FTIR spectrum of reed grass carbon after activation

The results of FTIR measurements of eed grass carbon before and after activation obtained peaks that appear indicate the presence of several functional groups.

Table 3. FTIR spectrum of reed grass Carbon before and after activation

Peak	Reed grass carbon before activation (cm^{-1})	Reed grass carbon after activation (cm^{-1})	Wavelength (cm^{-1})	Functional groups
1	3427,51	33908,6	3200-3600	O-H
2	1604,77	16067,0	1500-1600	C=C
3	1440,83	1440,83	1650-1750	C-H
4	1097,50	1099,43	1000-1300	C-O

The absorption of hydroxyl groups (O-H) indicates the presence of cellulose because hydroxyl is one of the main functional groups in the cellulose structure that can bind dyes. After the reed grass carbon sample was activated, there was an increase in intensity and a change in peak position indicating that there had been the formation of new functional groups.

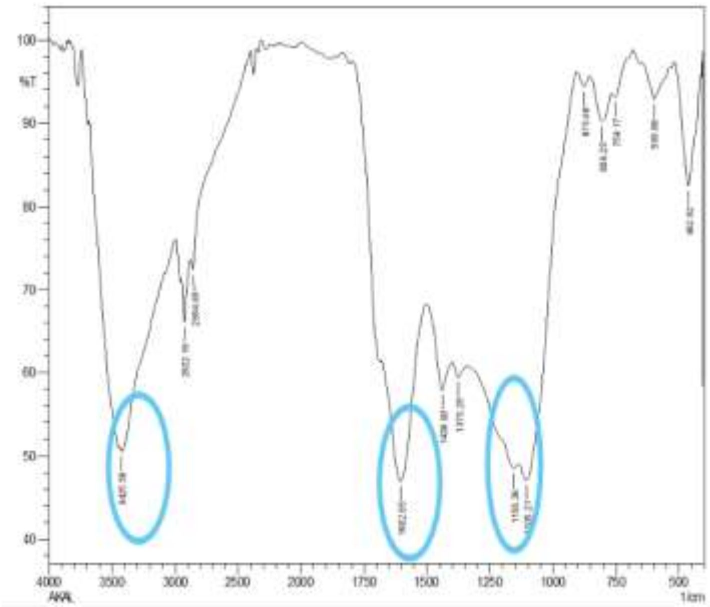
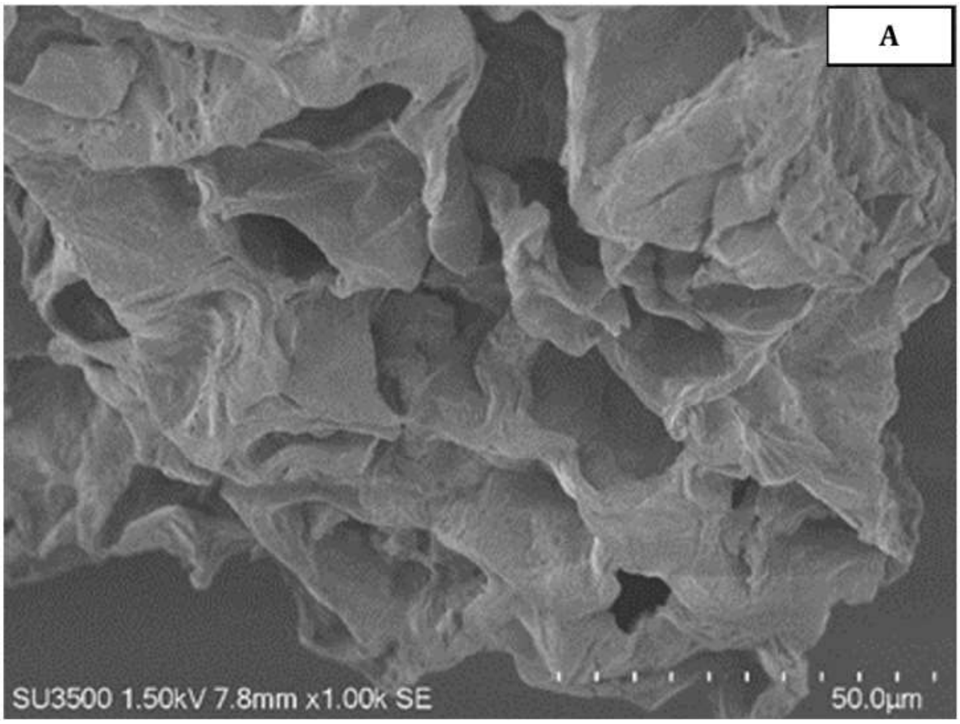


Figure 3. FTIR Spectrum of Activated Carbon After Adsorption

Based on the analysis, there are main peaks that show strong absorption characteristics in the areas of C-O (1105.21 cm^{-1}), N-H stretching (3425.58 cm^{-1}), and C=C stretching (1602.85 cm^{-1}). In activated carbon samples after adsorption, the transmittance value shows changes that can be attributed to the binding of adsorbate molecules on the surface of activated carbon.

SEM Characterization



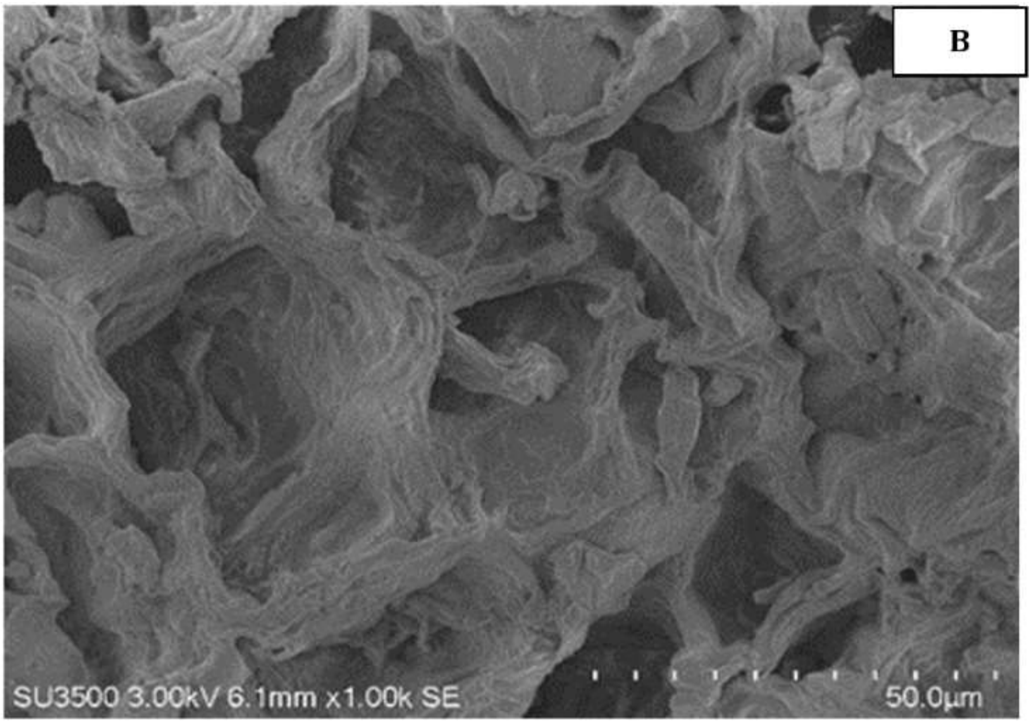
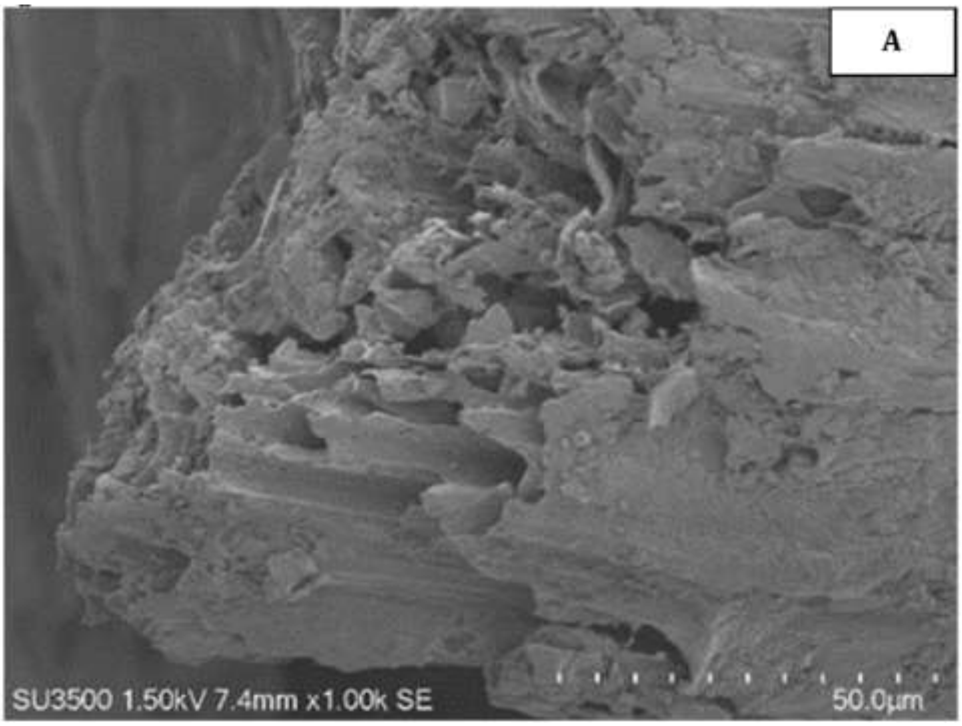


Figure 4. (A) SEM of coffee grounds carbon before activation (B) SEM of coffee grounds carbon after activation

Table 4. SEM of coffee grounds Carbon before and after activation

Treatment	Weight (%) Element						Porosity
	C	O	K	Mg	Al	Au	
Carbonization	79,18	18,69	-	0,56	0,47	1,09	0,574225
Activation	83,35	15,65	0,99	-	-	-	0,9971

SEM observations showed significant pores on the surface of robusta coffee grounds activated carbon, allowing it to adsorb more effectively.



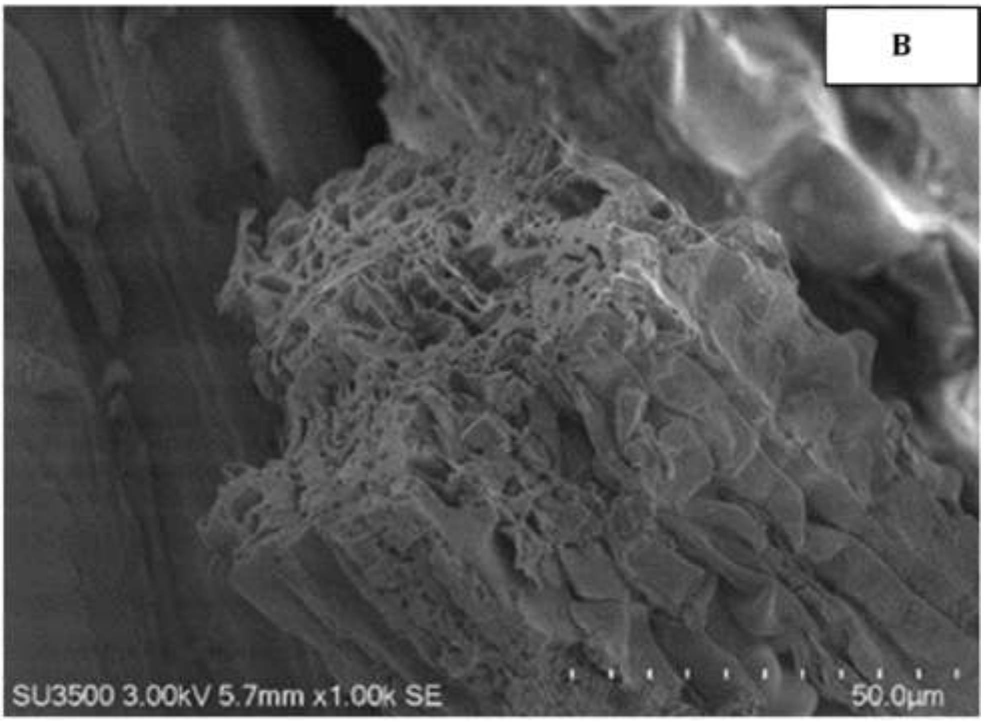


Figure 5. (A) SEM of reed grass carbon before activation (B) SEM of reed grass carbon after activation

Table 5. SEM of reed grass Carbon before and after activation

Treatment	Weight (%) Element						Porosity
	C	O	Al	Si	Au	K	
Carbonization	62,20	23,19	-	5,56	1,20	2,85	0,5978
Activation	59,79	32,49	0,61	7,10	-	-	0,9997

SEM observations show the presence of pores that are large enough and evenly distributed on the surface of reed activated carbon, making it more efficient in adsorbing.

Batch Method Adsorption

Figure 6 shows the curve of the effect of contact time variation on the % adsorption of methylene blue:

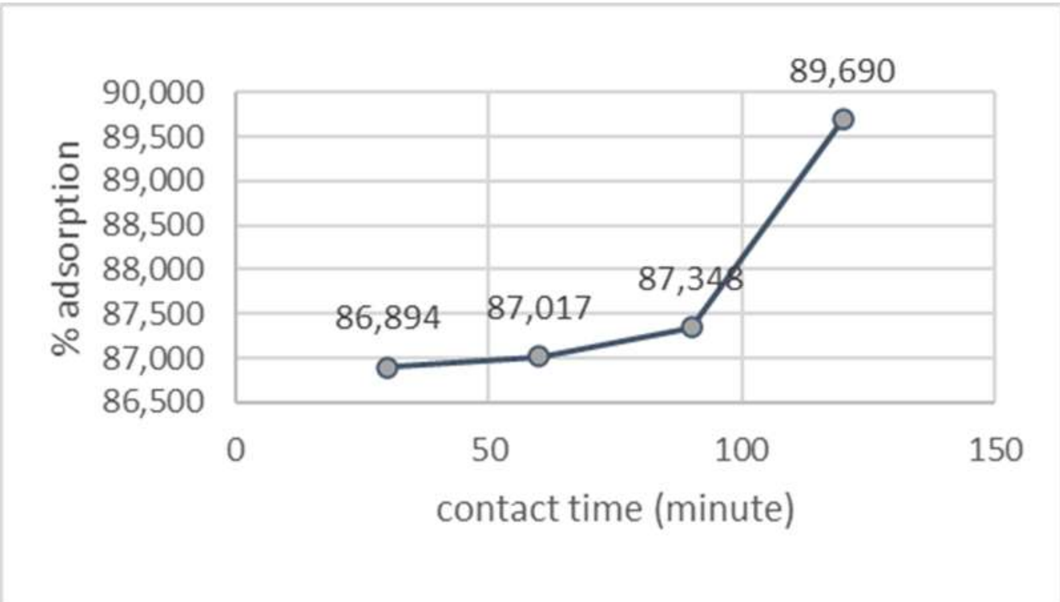


Figure 6. Effect Curve of Contact Time Variation on % Adsorption

Based on the results in Figure 6, it was found that the optimum contact time occurred at 120 minutes with a % adsorption of 89.69%. It was found that the longer the contact time, the more opportunities the particles of the adsorbent can intersect with the solution, resulting in the amount of methylene blue adsorbed will increase.

In this study, adsorption tests were carried out with variations in methylene blue concentration with constant time and mass (120 minutes and 0.1 gram). Figure 7 shows the curve of the effect of concentration variation on the % adsorption of methylene blue:

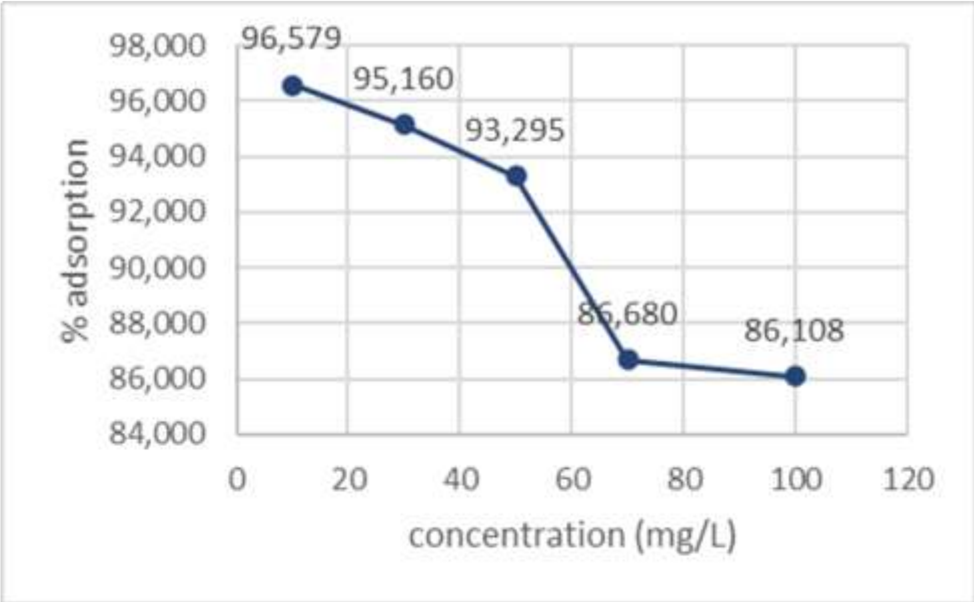


Figure 7. Curve of Effect of Concentration Variation on % Adsorption

Based on the results in Figure 7, it is obtained that the highest % adsorption is when the concentration is 10 ppm and the % adsorption is 96.579%. This is because the low concentration will provide an opportunity for all adsorbates to interact with the active groups of the bioadsorbent optimally. While at high concentrations, it can allow desorption.

In this study, adsorption tests were carried out with variations in the mass of coffee grounds and reed grass activated carbon bioadsorbents with an initial concentration and constant time (10 mg/L and 120 minutes). Figure 8 shows the curve of the effect of mass variation on the % adsorption of methylene blue:

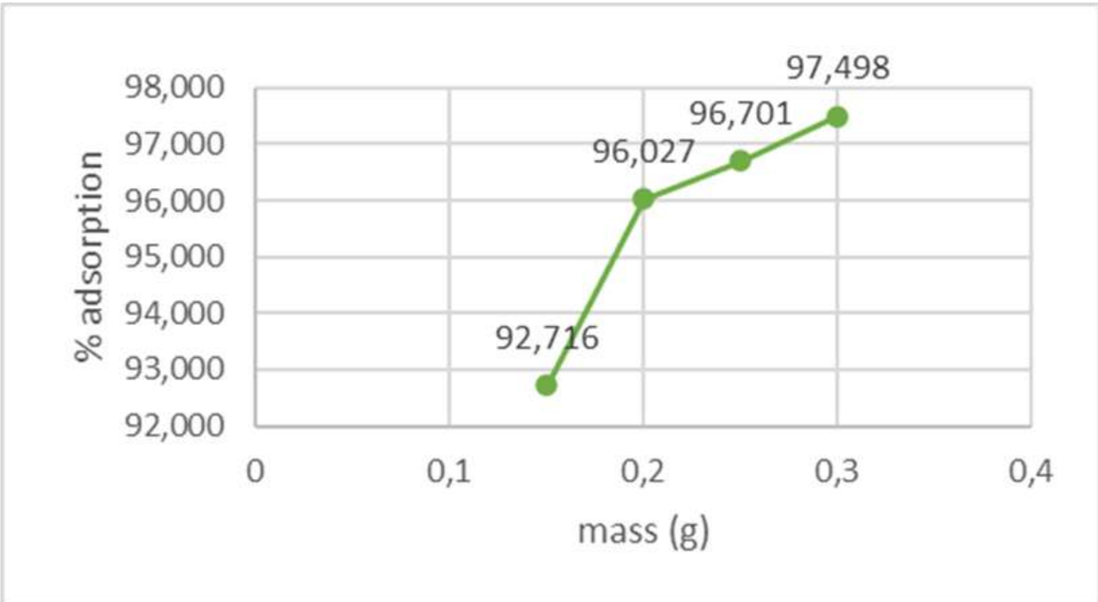


Figure 8. Effect Curve of Mass Variation on % Adsorption

Based on the results in Figure 8, the optimum mass is obtained at a mixed mass of 0.3 grams so that the % adsorption is 97.498%. The more mass of activated carbon bioadsorbent used, the greater the decrease in the final concentration of methylene blue. This is because when more mass is used, the number of bioadsorbent particles increases so that the opportunity for methylene blue to adhere to the bioadsorbent surface will be higher.

In this study, the adsorption capacity was determined using the Langmuir and Freundlich isotherm methods.

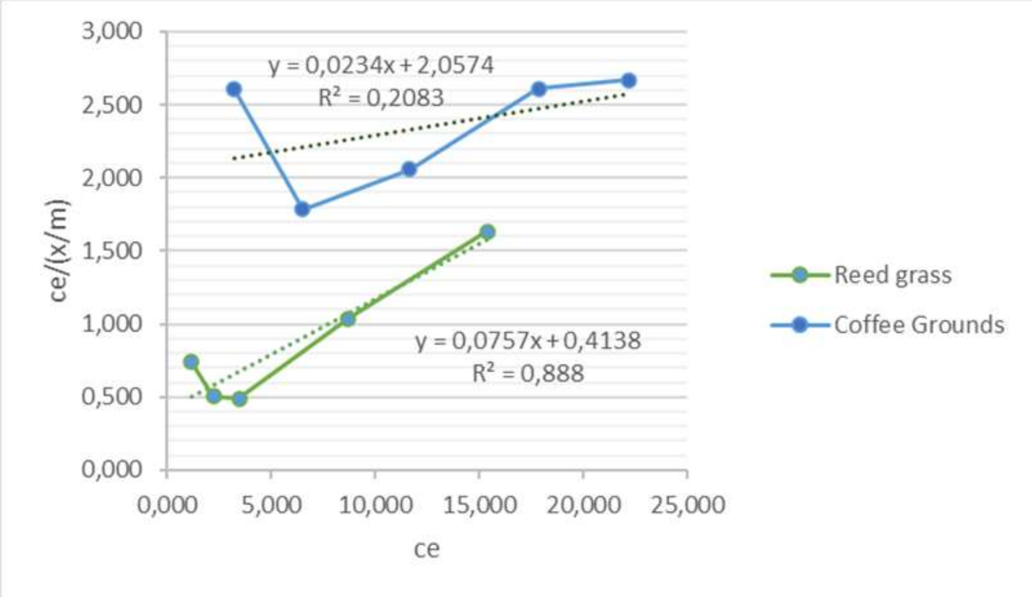


Figure 9. Langmuir Isotherm Curve of Coffee grounds and Reed grass

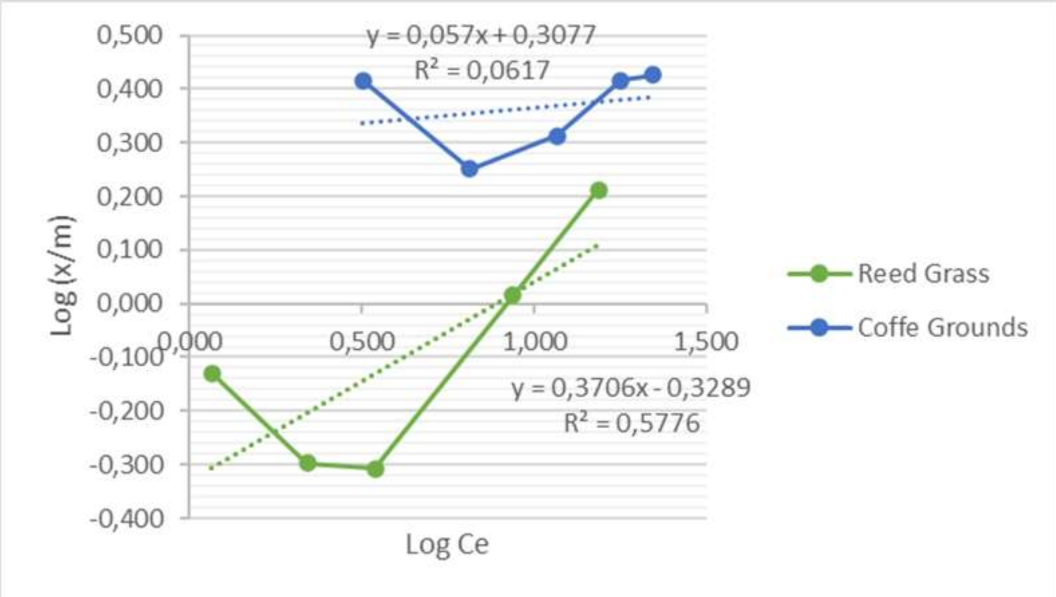


Figure 10. Freundlich Isotherm Curve of Coffee grounds and Reed grass

The appropriate isotherm type is determined based on the correlation coefficient value (R^2) which is close to 1. Based on the figure above, it is found that the method that better describes the methylene blue adsorption process is the Langmuir isotherm. This is evident from the resulting R^2 value of 0,888. Below is a table of constant values of Langmuir and Freundlich isotherms.

Table 6. Langmuir and Freundlich Isotherm Constant Values

Bioadsorbent	Isoterm	Constant	Value
Coffee Grounds	Langmuir	a (mg/g)	42,735
		b	0,011
	Freundlich	k (mg/g)	2,031
		n	17,544
Reed Grass	Langmuir	a (mg/g)	13,210
		b	0,183
	Freundlich	k (mg/g)	2,133
		n	2,698

Based on the results of calculations using the Langmuir isotherm equation, the maximum adsorption capacity of coffee grounds activated carbon was 42.735 mg/g and reed grass activated carbon was 13.210 mg/g. This shows that the adsorption process only forms a monolayer or one layer composed of adsorbate on the surface of the bioadsorben.

CONCLUSIONS

Based on the results of this study, activated carbon derived from robusta coffee grounds successfully met the SNI 06-3730-1995 standard, while activated carbon from reed grass did not fully satisfy the standard due to its volatile matter value of 50.24% and fixed carbon value of 43.46%. The optimum adsorption conditions for methylene blue removal in the batch system were obtained at a contact time of 120 minutes, an initial concentration of 10 mg/L, and a bioadsorbent mass of 0.3 g, resulting in an adsorption efficiency of 97.498%. Furthermore, adsorption isotherm analysis showed that the adsorption process was better described by the Langmuir isotherm model, with maximum adsorption capacities of 42.735 mg/g for coffee grounds activated carbon and 13.210 mg/g for reed grass activated carbon, indicating that the adsorption process predominantly occurred through monolayer adsorption on the bioadsorbent surface.

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