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Alkaline Modified *Archidendron jiringa* Peel as an Efficient Biosorbent for Methylene Blue Dye Removal

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ABSTRACT

Archidendron jiringa peel was investigated as a biosorbent for the removal of methylene blue (MB) dye from aqueous solutions. The raw Archidendron jiringa peel (RAJ) was modified using a sodium hydroxide solution to enhance its adsorption capacity. The modification process resulted in a cleaner and uniform surface on the peel, as revealed by SEM micrograph characterization. Batch experiments demonstrated that the chemically modified Archidendron jiringa peel (MAJ) exhibited higher adsorption capacity compared to the RAJ, attributable to the improved surface properties. Kinetic studies indicated that the adsorption process followed pseudo-first-order kinetics. Isotherm modeling revealed that the adsorption of MB conformed well to the Langmuir isotherm model, indicating monolayer adsorption. The maximum adsorption capacity increased from 22.09 mg g^{-1} for the RAJ to 42.18 mg g^{-1} for the MAJ. The adsorption capacity was influenced by pH, with higher adsorption observed at pH values above the point of zero charge (pHzpc), while it decreased with increasing biosorbent dosage due to adsorption site saturation. Overall, these results demonstrate the potential of NaOH-modified A. jiringa peel as an effective biosorbent for the removal of MB dye from wastewater.

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Keywords: Archidendron jiringa; Biosorption; Dye removal; Jering; Methylene Blue

INTRODUCTION

Textile dye wastewater poses a significant environmental challenge due to its high volume and composition of pollutants, including cationic dyes. These dyes are extensively used in various industries and can cause harm to the environment if not properly managed. The textile industry, which accounts for approximately 54% of the dye effluent, is the largest contributor to the significant volume of wastewater generated by the dye-producing and dye-utilizing industries globally, amounting to 3000-4000 kilotons [1]. Textile effluents contain a multitude of contaminants, such as dyes, surfactants, heavy metals, and other recalcitrant organic compounds [2]. Among these contaminants, cationic dyes, classified by their positive charge, including azo dyes, the largest group of colorants produced, pose a particular concern due to their versatility and widespread use [3]. As mentioned in the study by Berradi *et al.* [4], approximately 50% of these azo dyes fail to bind during the dyeing process, further worsening the pollution issues linked to textile dye wastewater.

Agricultural waste holds great promise as an alternative to conventional adsorbents due to its abundance, cost-effectiveness, and eco-friendliness. However, using raw, unmodified agricultural waste as a biosorbent poses challenges such as subpar performance and the release of organic compounds [5]. Consequently, researchers have focused on modifying agricultural waste to enhance its adsorption capacity [6]. Various methods, including the use of alkalis, acids, and surfactant-treated biomass, have been studied and demonstrated to be highly effective [7]. However, this study utilizes alkaline treatment instead of acid methods due to its superiority in reducing lignin, which is crucial for the adsorption process [8,9]. Moreover, alkaline treatment is cost-effective, widely available, and highly efficient based on previous research [10,11].

In this study, we utilized *Archidendron jiringa peel*, commonly known as Jering or Jengkol in Malaysia and Indonesia, as a natural material for the removal of methylene blue (MB) dye from wastewater. *A. jiringa* peel

was chosen as a biosorbent due to its affordability, easy availability, and possession of essential functional groups for effective biosorption. It has been observed to contain various functional groups, including phenols, amines, carbonyls, and carboxylic acids, as well as lignocellulose and hemicellulose [12,13]. Previous studies have also employed similar materials like jering or jengkol in different forms, such as unmodified, modified, and as a precursor for activated carbon, for pollutant removal [13-15]. The objective of this study is to investigate the effectiveness of NaOH-treated *A. jiringa* peel in removing cationic pollutants, such as Methylene Blue. Previous research utilizing alkaline treatment has shown that the surface properties of the peel of *Pithecellobium jiringa* can be modified, resulting in an increased surface area and exposure of functional groups [16].

METHODOLOGY

Materials

Methylene blue was obtained from Friendemann Schmidt (Parkwood, WA, US) (Fig. 1), and sodium hydroxide (NaOH) was purchased from R & M Chemicals (UK). Throughout the adsorption experiments, deionized water was used.

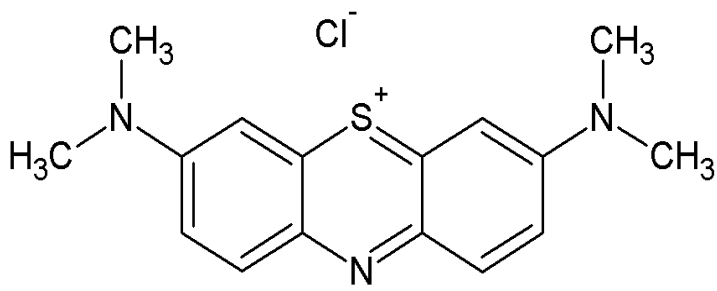

Figure 1: Chemical structure of MB.

Preparation of Biosorbent

The biosorbent was prepared by drying *A. jiringa* peel obtained from a local market in Klang, Malaysia, for about two days under sunlight, followed by shearing using a laboratory blender and sieving through a 100-mesh sieve. It was then washed with distilled water and dried in an oven at 60°C for 24 hours to obtain the final product. The dried and ground *A. jiringa* peel was stored in an airtight bottle and labeled as RAJ (Raw *A. jiringa* peel).

The RAJ was subjected to alkali modification using a 0.5 M sodium hydroxide (NaOH) solution for 24 hours at ambient temperature with stirring at 150 rpm. The alkaline treated *A. jiringa* peel was washed multiple times with distilled water until the wash water became clear. It was then dried in an oven at 60 °C for 24 hours to obtain the final product. The alkaline treated *A. jiringa* peel was abbreviated MAJ.

To prepare a stock solution of methylene blue (MB) dye wastewater, 0.5 g of MB powder was mixed with 1000 mL of water. This resulted in a concentration of 500 mg L^{-1}. Subsequently, the MB dye solution was diluted with deionized water to achieve the desired concentration.

Biosorbent Characterization

The surface morphologies of RAJ and MAJ were examined using a (Hitachi TM3030 Plus, Japan) scanning electron microscope (SEM) with an acceleration voltage of 5kV and a magnification of 500x. The pH point of zero charge (pHPZC) for RAJ and MAJ was determined using the salt addition method, following the procedure outlined in the study by [17].

Batch Experiments

Batch kinetic experiments were performed at room temperature to evaluate the adsorption capacity of RAJ and MAJ for MB dye removal from wastewater. In each experiment, 0.1 g of the biosorbent was mixed with 50 mL of MB dye solution of known concentration in a 125 mL Erlenmeyer flask, and the mixture was agitated for a pre-determined time. A control experiment without biosorbent material was also conducted to determine other factors' contribution to MB dye adsorption. All experiments were performed in duplicate under similar conditions, and the average

values were used for calculations. The amount of MB dye adsorbed by the biosorbent at time t, q_t (mg g^{-1}), was determined using the following Eq. (1):

$$q_t = \frac{C_o - C_t}{m} V \qquad (1)$$

where C_o and C_t are MB dye concentrations in mg L^{-1} initially and at time, t respectively, V is the volume of adsorbate in liter (L), and m is the weight of the biosorbent in gram (g).

A batch isotherm study was conducted to investigate the adsorption behavior of the biosorbent towards MB dye. The study involved mixing the biosorbent with the dye solution at five different biosorbent weights, ranging from 0.01 to 0.3 g, in order to determine the adsorption characteristics. The pH, concentration of MB dye and stirring speed were kept constant throughout the experiments. Once equilibrium was reached, a sample was withdrawn for analysis. The amount of MB dye adsorbed (q_e, mg g^{-1}) was determined using Eq. (2):

$$q_e = \frac{C_o - C_e}{m} V \qquad (2)$$

where C_o and C_e are MB dye concentrations in mg L^{-1} before and after adsorption, V is the volume of adsorbate in liter (L), and m is the weight of the biosorbent in gram (g).

Various experimental parameters, including biosorbent dosage and pH, were investigated to determine their effect on MB dye uptake while keeping other parameters constant. Samples were analyzed at equilibrium time to determine the remaining MB dye concentration. Throughout the experiment, an orbital shaker (Innova 2100 platform shaker) was used for agitation at 120 rpm. Dye concentration was measured using a DR2800 portable spectrophotometer at a wavelength of 667 nm, and pH was measured with a Mettler Toledo S20 pH Meter. The initial pH was 4.74, and 50 mL of MB dye solution was used. A biosorbent dosage of 0.1 g was applied while experiments were at room temperature.

RESULTS & DISCUSSION

Surface Characterization of RAJ and MAJ

It was obvious that before modification, the raw *A. jiringa* peel surface, as evidenced in the scanning electron microscope (SEM) micrograph in Fig. 2, was a rough and chaotic structure, on which many impurities were deposited, and holes and structures were filled with impurities. After modification, the surface of the modified peel (MAJ) became cleaner and more uniform due to most of the impurities being washed off, the breakdown of the fiber structure, and the elimination of lignin, wax, and oils as suggested by Mafa *et al.* [9] and Thakur *et al.* [18]. The increased space resulting from alkali modification enhances dye removal by providing more opportunities for dyes to be trapped or adsorbed, as observed by Song *et al.* [19] in their study on the removal of Cd^{2+} using NaOH-modified *Auricularia auricular* biosorbent.

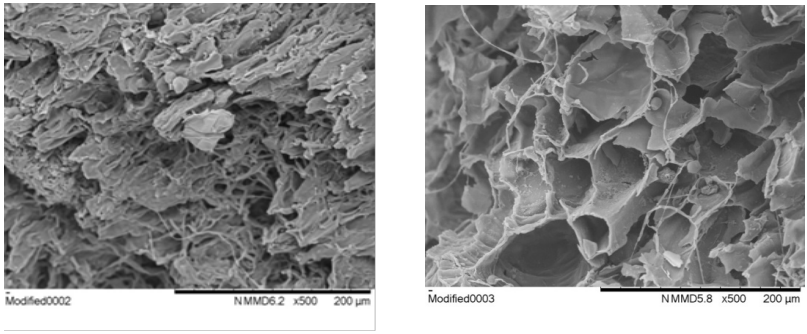

Figure 2: SEM micrographs of (a) RAJ and (b) MAJ

Adsorption Kinetic Experiments

The adsorption kinetics data were analyzed using the nonlinear pseudo-first-order and pseudo-second-order kinetic models, represented by Eq.(3) and (4), respectively:

$$q_t = q_e\left(1 - e^{-k_1 t}\right) \qquad (3)$$

$$q_t = \frac{q_e^2 k_2 t}{(1+q_e k_2 t)} \qquad (4)$$

In both equations, q_e represents the amount of MB adsorbed at equilibrium, q_t represents the amount of MB adsorbed at time t, and k_1 or k_2 are the rate constants for the respective models.

In Table 1 and Fig.3, the regression coefficient (R^2) values for the pseudo-first-order and pseudo-second-order kinetic models were above 0.95, indicating a good fit with the experimental data. However, while the pseudo-second-order model had a slightly higher R^2 value, indicating a better fit, the adsorption capacity results revealed a different outcome.

Table 1: Pseudo-first-order and pseudo-second-order rate constants.

	Kinetic Models						
Experiment		**Pseudo first order**			**Pseudo second order**		
	q_e, exp (mg g^{-1})	k_1 (min^{-1})	qe, cal (mg g^{-1})	R^2	k_2 (min^{-1})	q_e, cal (mg g^{-1})	R^2
RAJ	26.92	0.032	26.48	0.998	0.001	33.40	0.995
MAJ	32.46	0.139	32.63	0.979	0.009	34.43	0.990

The experimental adsorption capacity was 26.29 mg g^{-1} for RAJ and 32.46 mg g^{-1} for MAJ. Meanwhile, the predicted adsorption capacity using the pseudo-first-order model was 26.48 and 32.638 mg g^{-1}; for the pseudo-second-order model, it was 33.42 and 34.43 mg g^{-1}, respectively. The data showed that the qe values calculated for RAJ and MAJ, using the pseudo-second-order model, were significantly lower than the experimental values, while the q_e values calculated using the first-order kinetics model closely matched the experimental values. Based on these, the study suggests that the pseudo-first-order model provided a better description of the adsorption behavior of MB by both RAJ and MAJ, as it was more consistent with the experimental values. The pseudo-first-order model, which considers the adsorption process reversible and more suited to physisorption, was more applicable in this case. The limitations of using R^2 to describe adsorption kinetics were also observed by Jumasiah *et al.* [20]; where they indicated that while R^2 determination is valuable, adsorption capacity results provide a more definitive conclusion regarding the kinetic model fit.

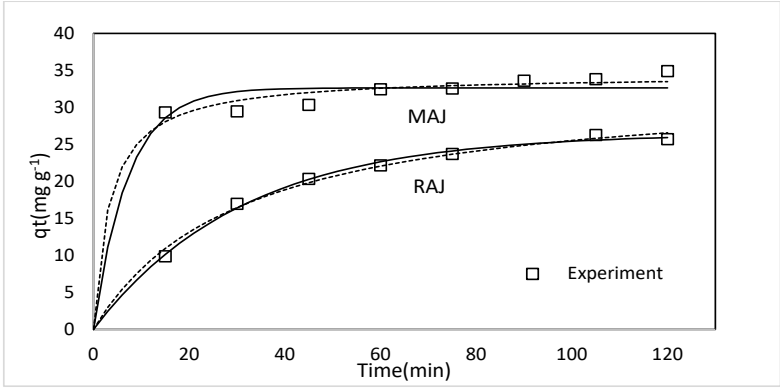

Figure 3: Adsorption kinetic models by nonlinear method for adsorption of MB on RAJ and MAJ

Adsorption Isotherm Experiments

The adsorption isotherm data were analyzed using the Langmuir and Freundlich models. The Langmuir and Freundlich model are given by Eq. (5) and (6), respectively:

$$q = \frac{Q_{max}}{1+bc_e} \qquad (5)$$

$$q = K_F {c_e}^{1/n} \qquad (6)$$

In both equations, Q_{max} represents the maximum adsorption capacity, C_e represents the concentration of the dye solution at equilibrium, and b or K_F are constants related to the energy or adsorption capacity, respectively. Additionally, the Freundlich model includes an empirical formula, n, which varies with the degree of heterogeneity.

Table 2 and Fig. 4 provide the fitting parameters obtained from modeling the measured isotherm data for the adsorption of MB onto RAJ and MAJ using the nonlinear forms of Langmuir and Freundlich models. These parameters are essential for accurately characterizing the adsorption behavior of MB on both RAJ and MAJ, enabling the development of reliable equations for design purposes. The Langmuir and Freundlich isotherm models exhibit a good fit, with nonlinear correlation coefficients (R^2) of

over 0.9 for both RAJ and MAJ. However, Langmuir demonstrated a better fit with R^2 of 0.919 and 0.969 for RAJ and MAJ, respectively, compared to Freundlich. The Langmuir isotherm model indicates that there was monolayer sorption and uniform distribution of active sites on the surface of the biosorbent. The monolayer capacity (Q_{max}) for MB adsorption at 313 K for RAJ and MAJ are 22.09 and 42.18 mg g^{-1}, respectively, suggesting that the NaOH treatment improved the removal capacity. The Freundlich model assumes that the uptake of MB occurs on a heterogeneous biosorbent surface, and the value of the Freundlich exponent, n (>1), indicates a favourable adsorption process, as suggested by Foo and Hameed [21]. The values of n for both biosorbents were found to be favourable at 6.02 and 3.90 for RAJ and MAJ, respectively.

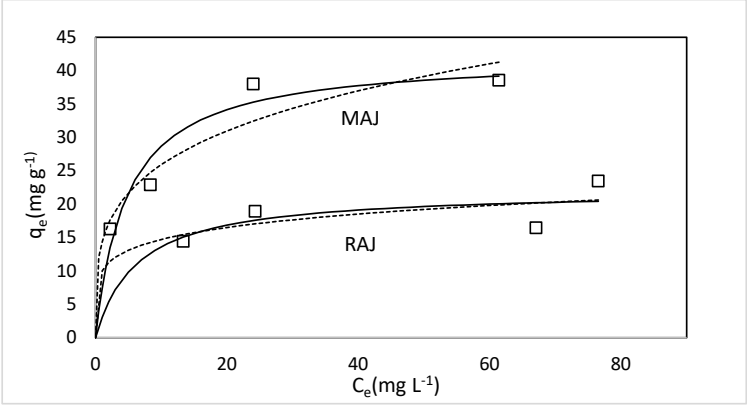

Figure 4: Adsorption isotherms by nonlinear method for adsorption of MB onto RAJ and MAJ

Table 2: Freundlich and Langmuir isotherm constants for MB sorption

Biosorbent	Langmuir			Freundlich		
	Q_{max} (mg g^{-1})	b (L mg^{-1})	R^2	K_F (L g^{-1})	n	R^2
RAJ	22.09	0.16	0.92	10.04	6.02	0.91
MAJ	42.18	0.21	0.97	14.37	3.90	0.95

Adsorption Equilibrium Experiments

The adsorption capacities of RAJ and MAJ for MB dye removal were examined at various pH levels (2, 5, 7, and 9), with the corresponding pHzpc values measured as 7.03 and 7.23 for RAJ and MAJ, respectively, indicating neutral surfaces for the adsorbents. Below the pHzpc, a positive charge was observed, while above it, a negative charge was present [22]. Figure 5 demonstrates that MAJ exhibited higher dye removal capacities compared to RAJ. At pH 2, RAJ achieved a removal capacity of 13.5 mg g^{-1}, whereas MAJ reached 15.8 mg g^{-1}. Similarly, at pH 5, 7, and 9, RAJ exhibited removal capacities of 26.3, 29.3, and 31.1 mg g^{-1}, respectively, while MAJ displayed capacities of 38.9, 40.4, and 46.0 mg g^{-1}. The removal efficiency of MB for both RAJ and MAJ was observed as higher above their respective pHzpc values. Pirbazari *et al.* [23] reported that the adsorption of the cationic dye MB is favored at pH levels higher than the pHzpc due to the presence of functional groups like OH- and COO- groups. This facilitates increased adsorption of MB molecules onto the biosorbent surface, enhancing removal efficiency. Numerous studies have reported the impact of pH on MB removal utilizing various biosorbents. For example, Yagub *et al.* [24] found that both raw and alkaline-treated pine cone powder biosorbents exhibited significantly higher MB removal at pH solution of above pHzpc.

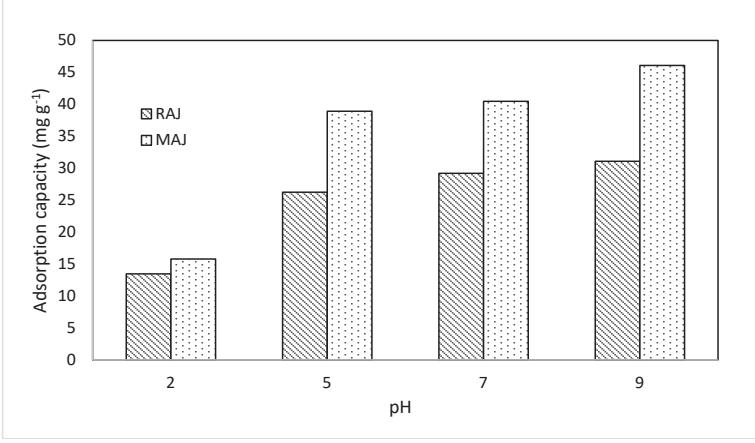

Figure 5: Effect of pH on adsorption of MB onto RAJ and MAJ

The removal capacity of MB dye from aqueous solutions using RAJ and MAJ at different dosages (0.2, 1, 4, and 6 g L^{-1}) was investigated. The findings, depicted in Figure 6, indicate that the removal capacity of MB generally decreased with increasing dosage for both RAJ and MAJ. This observation is in line with previous studies [25]. The highest removal capacity of MB dye using RAJ was observed at a dosage of 0.2 g L^{-1}, where 35.68 mg g^{-1} of dye was removed. However, as the dosages increased to 1, 4, and 6 g L^{-1}, the removal capacity decreased to 23.45, 16.47, and 18.92 mg g^{-1}, respectively. This finding, consistent with earlier research on canola residues biosorbent for MB removal [26], indicates that the adsorption sites on the surface of biosorbent became saturated at higher dosages, resulting in a decline in the removal capacity. In contrast, MAJ exhibited the highest removal capacity at a dosage of 0.2 g $L^{-1,}$ removing 75.32 mg g^{-1} of dye. As the dosage increased to 1, 4, and 6 g L^{-1}, the removal capacity decreased to 38.59, 37.99, and 16.30 mg g^{-1}, respectively. The enhanced removal capacity of MAJ can be attributed to the alkaline modification, as suggested by Oyelude and Appiah-Takyi [27] in their study, where the treatment of sorghum mash with NaOH improves its surface properties, resulting in higher removal capacity at lower dosages.

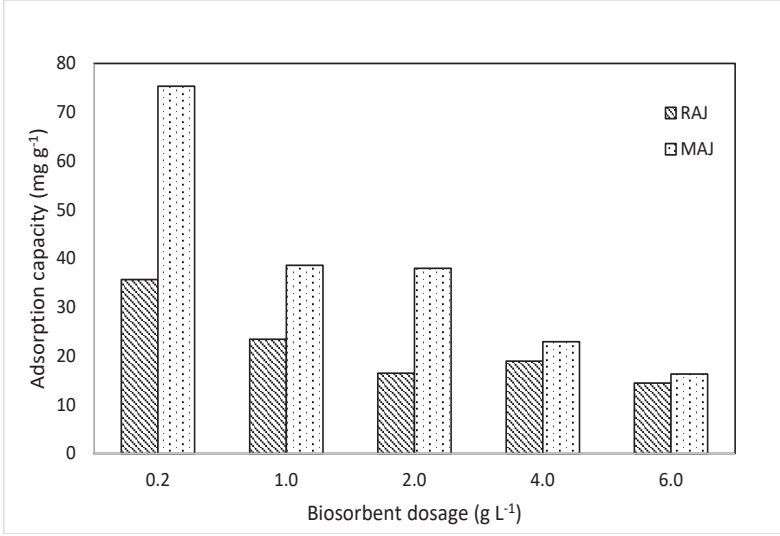

Figure 6: Effect of dosage on adsorption of MB onto RAJ and MAJ

CONCLUSION

The alkali modification of *Archidendron jiringa* peel using sodium hydroxide successfully improved its adsorption capacity for methylene blue dye removal. The modification process resulted in a cleaner and more uniform surface on the peel, as revealed by characterization of SEM micrograph. Kinetic studies indicated a pseudo-first-order adsorption pattern, suggesting physisorption as the predominant mechanism. Isotherm modeling analysis showed that the experimental data fitted well with the Langmuir model, indicating monolayer adsorption. The maximum adsorption capacity significantly increased from 22.08 mg g^{-1} to 42.18 mg g^{-1}, indicating the effectiveness of the alkali treatment in enhancing the peel's adsorption performance. The adsorption capacity was found to be influenced by pH, with higher adsorption observed at pH values above the point of zero charge (pHzpc) due to stronger electrostatic attraction between the dye and the modified peel. However, the adsorption capacity decreased with increasing dosage, likely due to site saturation at high concentrations. The alkali-modified peel demonstrated its potential as an eco-friendly and cost-effective biosorbent for methylene blue removal. Overall, the alkali treatment effectively enhanced the adsorption capacity of the peel by increasing its surface area and exposing more functional groups, thus improving its performance as a biosorbent compared to the raw *Archidendron jiringa* peel.

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