

Study on Adsorption Kinetics of Methylene Blue by Modified Sago Frond Waste

Kajian Kinetika Adsorpsi Metilena Biru oleh Limbah Pelepah Sagu Termodifikasi

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ABSTRACT

The modified sago frond waste (PSM) using the Fast Microwave-Assisted Acid method, which has several characteristic changes, was tested to determine its adsorption ability to a cationic textile dye. PSM samples as adsorbents have variations in the modification of oxalic acid addition at 0; 1,5; 3,0 and 4,5% (w/v) were used for the adsorption of Methylene Blue (MB) dye on several parameters. This study was conducted to determine the adsorption kinetics through the effect of the ratio of the adsorbent and the contact time and the acid variation of the adsorbent as additional variables. Optimum MB absorption was obtained at a ratio of 0,6 g/L and a contact time of 120 minutes. The data results were analyzed using four general kinetic models: pseudo-first-order, pseudo-second-order, intra-particle diffusion, and Elovich equation. Adsorption followed the pseudo-second-order reaction rate with a coefficient of determination (R2) 0.9996-0.9999 in all variations of PSM. The theoretical adsorption capacity was 25.58 to 27.32 mg/g, and the effect of increasing acid on PSM increased the adsorption and absorption capacity of MB.

Keywords: sago frond waste, adsorption, kinetic models, methylene blue.

ABSTRAK

Limbah pelepah sagu hasil modifikasi (PSM) dengan metode Fast Microwave-Assisted Acid yang memiliki beberapa perubahan karakteristik diuji untuk mengetahui kemampuan adsorpsinya terhadap suatu zat warna tekstil kationik. Sampel PSM sebagai adsorben memiliki variasi modifikasi penambahan asam oksalat pada 0; 1,5; 3,0 dan 4,5% (b/v) digunakan untuk adsorpsi zat warna Metilena Biru (MB) pada beberapa parameter. Studi ini dilakukan untuk mengetahui tinjauan kinetika adsorpsi melalui pengaruh dari rasio adsorben dan waktu kontak serta variasi asam dari adsorben juga menjadi variabel tambahan. Penyerapan optimum MB didapat pada rasio 0,6 g/L dan waktu kontak 120 menit. Untuk memperkirakan model kinetika adsorpsi MB pada PSM, empat model kinetika umum yaitu orde pertama semu, orde kedua semu, difusi intra-partikel dan persamaan Elovich telah digunakan . Adsorpsi diketahui mengikuti laju reaksi orde kedua semu dengan tingkat koefisien determinasi (R²) 0,9996-0,9999 pada semua variasi PSM. Kapasitas adsorpsi maksimum teoritis diketahui sebesar25,58 hingga 27,32 g/mg dan peningkatan asam pada PSM juga diketahui dapat meningkatkan kapasitas adsorpsi serta penyerapan dari MB.

Kata Kunci: pelepah sagu, adsorpsi, model kinetika, metilena biru.

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1. INTRODUCTION

The continuous development of the industry has led to a massive increase in the release of excess synthetic dyestuffs into water sources. According to Mittal et al. (2010), Saleh and Gupta (2014), and Anwar and Mulyadi (2015), more than 15% of synthetic dyestuffs are lost or separated during the manufacturing and operational processes as industrial waste. Many industries waste contains dyes as a by-product of the process. This dye is present as a pollutant that is difficult to degrade naturally, has a high level of toxicity, and is carcinogenic (Oladipo et al., 2014). Industries that produce plastics, printing, cosmetics, rubber, leather, pharmaceuticals, food, and textiles are the most dominant industries that produce dye waste during their operations (Mittal et al., 2009). The ability of dyes to spread quickly causes these pollutants to change the color of polluted water easily. When dissolved in water, the dye will block sunlight from penetrating the water's surface and inhibit the photochemical and biological activity of the aquatic life. Therefore, removing or reducing synthetic dyes as contaminants in wastewater is of particular concern (Malik et al., 2007, Balarak et al., 2015).

Dyes are generally classified into cationic and anionic. Cationic dyes are more toxic than anionic dyes. One example of a cationic dye is methylene blue (C₁₆H₁₈N₃SCl, C.I. No. 52015). This dye is generally applied as dyes for cotton, wool, silk, leather, and coatings for paper stocks (Ghaedi et al., 2014). Although methylene blue is not very dangerous, longterm exposure could cause serious health problems such as increased heart rate, vomiting, shock, cyanosis, jaundice, quadriplegia, mental confusion, eye burns, tissue necrosis, and methemoglobinemia (Rafatullah et al., 2010, Kushwaha et al., 2014).

Removing contaminants from synthetic dyes is known to be quite tricky because of its resistance to aerobic and anaerobic decomposition. It is also stable to light, heat, and moderate oxidizing agents (Crini, 2006, Vimonses et al., 2009). One of the most effective technologies for removing dyes from waste is the adsorption process (Demirbas, 2009). Although the activated carbon adsorption method has been proven to be the most effective adsorbent (Mall et al., 2005, Namasivayam and Kavitha, 2002), its widespread application is hampered by the relatively high production cost of up to \$1.3-20/kg (Gupta et al., 2009). Therefore, various studies have been conducted to obtain other alternatives to develop low-cost adsorbents using lignocellulosic biomass and natural minerals (clay, zeolite).

Modified sago frond (PSM) has several changed characteristics, such as darker color, reduced adsorbent mass, reduced lignin composition, and changes in the crystallinity of the lignocellulosic structure (Tirani et al., 2021). This research was conducted to determine the adsorption capacity of the modified biomass source, especially in terms of its kinetics. In addition, the research results are expected to provide deeper а understanding of the MB adsorption mechanism on PSM.

2. MATERIALS AND METHODS 2.1. Materials

The materials used were Methylene Blue (C₁₆H₁₈CIN₃S, CAS.NO. 61-73-4) from PUDAK SCIENTIFIC and PSM samples from previous studies. The tools used were a series of glassware, Fujitsu FSR-A Analytical balance, UV-Visible spectrophotometer LGS 53 BEL® Photonics., and Orbital Shaker KJ-201BD OREGON. This research was conducted at the Forestchem Workshop, Banjarbaru.

2.2. Methods

The initial preparation was crushing and milling the sago frond into powder and small pieces, then drying in direct sunlight. The dried samples were filtered to a 40-60 mesh size and dried in an oven at 105°C for 30 minutes and then stored in an airtight container at room temperature. A total of 3

grams of prepared sago frond were added with water as a solvent; oxalic acid 0; 1.5; 3.0; & 4.5 % (w/v) with a 1:25 ratio of sample powder and solvent. The sample was then irradiated in a microwave with a power of 450 W for 10 minutes. After completion, the samples were rinsed and neutralized. The neutral sample was then placed in an oven at 105° C for 5 hours. This procedure was carried out in the previous studies (Tirani et al., 2021).

The 1000 ppm MB stock solution was prepared by dissolving 0.5 grams of MB in 500 mL of deionized water. MB standard solution was prepared by diluting stock solution up to 2; 3; 4; 5; 6, and 7 ppm. Determination of the maximum wavelength was carried out by measuring the absorption of 4 ppm MB solution in the range of 600-700 nm. The absorption of each standard solution was measured, and a calibration curve was made to determine the final concentration of the MB sample.

The first test parameter was the effect of PSM ratio (0.2; 0.3; 0.4; 0.5 and 0.6 g/L). PSM adsorbent with oxalic acid variation 0; 1.5; 3.0 and 4.5% of the previous study were taken (4; 6; 8; 10 and 12 mg), and a 20 ppm MB solution was prepared from the dilution of the stock solution, respectively. A total of 20 mL of the MB solution was put into an Erlenmeyer, and PSM adsorbent was added and then stirred with an Orbital Shaker for 120 minutes at 120 rpm. The adsorbed MB solution was taken, and the absorption was measured at a wavelength of 665 nm. The

second parameter was the effect of contact time (10, 20, 40, 80, and 120 minutes). A total of 12 mg PSM of all variations was tested at 20 mL MB 20 ppm at a predetermined time range, and the final absorption was measured. The adsorption and adsorption capacities of MB were then calculated.

3. RESULTS AND DISCUSSION

Modification of the biomass source (sago frond waste) was carried out using the Fast Microwave-Assisted Acid method. Samples of sago frond with variations in oxalic acid (0; 1.5; 3.0 and 4.5%) were irradiated by microwave at 450 W for 10 minutes. This pretreatment method has been used in previous studies, where the lignocellulosic structure was crushed to release cellulose and remove hemicellulose and lignin components (Tirani et al., 2021). The modified adsorbent was then used for the MB adsorption test by taking into account the adsorption capacity and percent absorption of MB, which was calculated through the following equation:

$$q_e = \frac{(C_0 - C_e) \times V}{m} \tag{1}$$

% absorption =
$$\frac{(C_0 - C_e)}{C_0}$$
 (2)

Where qe as adsorption capacity (mg/g), C0 and Ce as initial concentration and final concentration/equilibrium (mg/L), V as volume MB (L), and m is the mass of adsorbent used (g). PSM adsorbent is showed in Fig. 1.



Figure 1. PSM adsorbent (Tirani et al., 2021).

	Ratio (g/L)	Contact	Initial	Final Concentration (ppm)				
Parameter		Time (minute)	TimeConcentrationminute)(ppm)		1,5%	3,0%	4,5%	
Effect of	0.2	120	20	10.91	10.41	10.16	9.92	
adsorbent	03	120	20	9.08	8.71	8.50	8.35	
ratio	0.4	120	20	7.12	6.83	6.58	6.38	
	0.5	120	20	5.06	4.82	4.58	4.48	
	0.6	120	20	4.40	4.14	3.99	3.85	
Effect of	0.6	10	20	7.74	7.71	7.69	7.66	
contact time	0.6	20	20	6.96	6.73	6.41	6.64	
	0.6	40	20	6.19	5.54	5.08	4.84	
	0.6	80	20	5.45	4.83	4.48	4.24	
	0.6	120	20	5.00	4.72	4.44	4.13	

Table 1. MB concentration data before and after the adsorption

3.1. Effect of PSM Adsorbent ratio on adsorption ability for MB

The effect of the ratio and variation of acid on the adsorption and absorption capacity is presented in Fig. 2. These results indicate that the percentage of MB adsorption increases with the increase in the ratio of the adsorbent used. However, as it approaches 0.6 g/L, there is a smaller increase than before, which is the approach predicted to maximum absorption for the following parameters. An increase in the ratio also means an increase in the amount of adsorbent used. Therefore, the surface site for adsorption also increases, and the interaction between adsorbent-adsorbate increases the absorption percentage. Meanwhile, the adsorption capacity showed a decrease as the ratio increased. According to El Qada et al. (2006), increasing the

amount of adsorbent while maintaining the same volume and concentration of MB may

take longer to attain the maximal adsorption capacity. The sample with a lower ratio has more adsorption sites and thus a larger adsorption capacity. In addition, other studies also showed similar results when using activated carbon from corn husks (biomass) as MB adsorbent. According to Khodaie et al. (2013), decreased adsorption capacity at higher ratios could occur for two reasons. First is the unsaturation of the adsorption site that occurs during the adsorption process. Second, particulate interactions such as aggregation at high ratios lead to a decrease in the surface area of the adsorbent and an increase in the length of the diffusion path. Overall, the effect of acid variations on the adsorbent indicates that the increase in the concentration of oxalic acid used will increase the adsorption ability of PSM. The highest absorption occurred at 4.5% PSM adsorbent with a ratio of 0.6 g/L, 80.76% with an adsorption capacity of 26.92 mg/g.



Figure 2. Effect of adsorbent ratio on MB absorption and adsorption capacity (C0 = 20 ppm, contact time = 120 minutes, stirring speed = 120 rpm).



Figure 3. Effect of contact time on MB absorption and adsorption capacity (C0 = 20 ppm, adsorbent ratio = 0.6 g/L, stirring speed = 120 rpm).

3.2. Effect of contact time on adsorption ability for MB

By optimizing the previous adsorbent ratio, an adsorbent ratio of 0.6 g/L was taken

to maximize the absorption of MB. Fig. 3 illustrates the effect of the contact time for the adsorbent-adsorbate interaction on the adsorption and adsorption capacity.

In general, the pattern of increasing adsorption capacity with MB absorption is nearly identical to the trend of increasing contact time. A significant increase occurred at a time of up to 40 minutes, and after that, the increase was relatively small. This nottoo-significant increase can be indicated as a time towards equilibrium. Judging from the effect of the PSM acid variation itself, as the concentration of the acid used increases, it can accelerate the adsorption process to its equilibrium state and also slightly increase the adsorption capacity at the equilibrium state.

3.3. Kinetic Model

Determination of the appropriate model is needed to determine the adsorption mechanism. Several models that can be used are pseudo-first-order, pseudo-second-order (Lagergren, 1898), intra-particle diffusion (Ho and McKay, 1998), and the Elovich equation (Wu et al., 2009). The four models are based on Table 2 below.

Model	Equation		Plot		
pseudo-first-order	$\ln(q_{e,exp} - q_t) = \ln q_e - k_1 t$	(3)	$ln\left(q_{e,exp}\text{-}q_t\right)vs \ t$		
pseudo-second-order	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$	(4)	t/q _t vs t		
intra-particle diffusion	$q_t = k_{id}t^{1/2} + C$	(5)	$q_t \ vs \ t^{1/2}$		
Elovich equation	$q_t = \left(\frac{1}{\beta}\right) \ln(\alpha\beta) + \left(\frac{1}{\beta}\right) \ln(t)$	(6)	$q_t vs ln(t)$		

Table 2. Kinetic model of reaction rate mechanism

The model validity can be seen from each plot. The most suitable model is determined from the correlation coefficient (r) and/or the coefficient of determination (R2). Each model has its variable parameters such as qe,exp, namely adsorption capacity at experimental equilibrium (mg/g), qt, namely adsorption capacity at time t (mg/g), qe, namely theoretical adsorption capacity (mg/g), k_1 and k_2 are the rate constants for the pseudo-first-order (1 min⁻¹) and pseudo- $(g.mg^{-1}.min^{-1})$ second-order reactions. respectively. For the diffusion model, kid is the rate constant of the intra-particle diffusion model (mg.g⁻¹.min^{-1/2}), and C represents the thickness of the boundary layer (mg/g). For the Elovich equation, a represents the chemosorption rate at zero coverage (mg.g-¹.min⁻¹) and b corresponds to the level of surface coverage and the chemosorption activation energy (g/mg).

Through the plot graphs of the four models, the coefficient of determination (R²), normalized standard deviation (Dq), and sum squared errors (SSE) will become the reference for determining the most suitable model. Among all the types of kinetic models tested, the pseudo-second-order had the highest coefficient of determination, namely 0.9996-0.9999. Besides that, the Dq value of the model ranged from 0.019-0.044%. Another similar study was by Foo and Hameed (2012), which used industrial solid waste prepared by microwave for MB adsorption. The adsorption kinetics are also in line with the

pseudo-second-order model. The parameters obtained from this kinetic model are theoretical adsorption capacity which increases as the concentration of oxalic acid increases from the previous pretreatment process. This value was calculated at 25,576; 26,178; 26.667 and 27.322 mg/g with a pseudo-second-order reaction rate constant of 0.011135; 0.011854; 0.012133 and 0.010034 (1 min⁻¹) respectively. The adsorption capacity shown in the pseudo-second-order model is also close to the experimental adsorption capacity value, compared to the pseudo-first-order model, which has a significant difference in the value of the theoretical adsorption capacity to the experiment. Therefore, the mechanism that occurs during the adsorption of MB by PSM based on a pseudo-second-order kinetic model explains the presence of а corresponding chemosorption by sharing and exchanging electrons between MB and the PSM structure (Ho and McKay, 1998). Although not as good as the pseudo-secondorder, the pseudo-first-order kinetic model also shows a relatively high fit to the experimental data. The mechanism that occurs based on this model is adsorption which is influenced by the adsorbate so that the interactions that occur are limited to physical adsorption (Foo and Hameed, 2012).

The fundamental difference between the two Lagergren models lies in their basic properties, namely physisorption for pseudofirst-order and chemisorption for pseudosecond-order (Lagergren, 1898). As a conclusion from the consensus between the experimental data and the model, the mechanism that occurs during MB adsorption by PSM based on a pseudo-second-order kinetics model explains the existence of corresponding chemisorption by sharing and exchanging electrons between MB and the PSM structure (Ho and McKay, 1998), but does not mean that adsorption is physically absent or not involved in the process. Chemisorption (adsorption) can be assumed to act more dominantly as a rate-determining step of the reaction.

DSM a	a	Pseudo-first0order				Pseudo-second-order					
(b/v)	(mg/g)	$k_1 (1/sec)$	q _{e,cal}	P ²	Da(%)	k_2 (1/sec)	q _{e,cal}	h (mg²/	P ²	Dq	
$(0, v)$ $(\operatorname{Ing} g)$	KI (1/See)	(mg/g)	g) K	Dq (70)	K ₂ (1/300)	(mg/g)	g ² sec)	К	(%)		
0%	25.002	-0.0254	5.634	0.9977	0.086	0.011135	25.576	7.283	0.9996	0.044	
1.5%	25.463	-0.0485	8.676	0.9975	0.167	0.011854	26.178	8.124	0.9999	0.019	
3.0%	25.934	-0.0626	11.226	0.9967	0.247	0.012133	26.667	8.628	0.9999	0.020	
4.5%	26.449	-0.0505	10.111	0.9953	0.255	0.010034	27.322	7.491	0.9998	0.027	

 Table 3. Comparison of pseudo-first-order and pseudo-second-order kinetic model parameters of MB adsorption by several variations of PSM

Table 4. Comparison of the parameters of the intra-particle diffusion kinetics model and the Elovich equation

PSM	Difusi intra-partikel				Persamaan Elovich			
(b/v)	C (mg/g)	kid	\mathbb{R}^2	Dq	а	b	\mathbb{R}^2	Dq
0%	19.02	0.5711	0.9695	5.850	12747.49	0.545167	0.9999	0.986
1.5%	19.179	0,.6364	0.8941	63.638	4065.815	0.476872	0.9708	21.001
3.0%	19.411	0.673	0.8429	16.775	2518.834	0.444425	0.9423	32.141
4.5%	19.013	0.7604	0.8601	17.708	971.494	0.396715	0.9450	35.100



Figure 5. Comparison of pseudo-first-order plots (a) and pseudo-second-order plots (b)



Figure 6. Intra-particle diffusion kinetics model plot (a) and Elovich equation (b)



Figure 7. Curves characteristics based on the dimensionless Elovich equation

Another graph also exhibits samples with a kinetic model that matches the Elovich equation, namely 0% PSM. The Elovich equation describes a reaction involving the adsorption of a chemical substance on a solid surface without any product desorption. The reaction rate decreases with time due to increased surface coverage (Wu et al., 2009). Characteristics that can explain the type of reaction that occurs are indicated by the RE value of the equation:

$$\left(\frac{q_t}{q_{ref}}\right) = R_E \ln\left(\frac{t}{t_{ref}}\right) + 1 \tag{7}$$

The RE value at 0% PSM, which was 0.185, is in zone III, which ranges from 0.02 to 0.1, and this type of reaction means a rapid rise. According to Elovich's equation, the majority of adsorption systems are in zone II, while zones I and IV are very few (Wu et al., 2009). The intra-particle diffusion plot has a relatively low fit with the experimental data (except 0% PSM). With some of the available data, it can be assumed that the effect of chemisorption on determining the rate of a reaction is greater than that of physisorption or diffusion.

4. CONCLUSIONS

The experimental results of the treatment of sago frond by increasing the concentration of oxalic acid could increase the adsorption and absorption capacity of MB. The optimal adsorption process occurred at an adsorbent ratio of 0.6 g/L for 120 minutes, focusing on the highest MB absorption of 79.346% at 4.5% PSM with an adsorption capacity of 26.449 mg/g. The experimental data follow the pseudo-second-order kinetic model based the dominant chemosorption on as determining the reaction rate. This process has the advantage of fast and efficient adsorption.

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