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Removal of Procion Red MX-5B from aqueous solution by adsorption on *Parkia speciosa* (stink bean) peel powder

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ABSTRACT

The potential of *Parkia speciosa* peel (SBP) for removal of procion red mx-5B (PR) through the adsorption process was investigated. PR is a type of azo dye which is toxic to the environment especially in water. Sustainable adsorbents such as agricultural wastes have been promising to reduce the amount of pollution in wastewater due to their accessibility and more economic. The batch study was performed to examine the influence of varies exploratory conditions by fluctuating the dosage, adsorption time, and initial concentration. The main functional groups involved in the dye removal were analysed by Fourier transform infrared spectrometry (FTIR). The isotherm data were examined by the Langmuir, Freundlich and Temkin models. The result showed the equilibrium studies were perfectly associated by the Freundlich isotherm with R²= 0.9993, 0.9786 and 0.9728 for 1, 3 and 5 g of adsorbent dosage that corresponded with pseudo-second-order at a rate constant of 1x10⁻³/min. The FTIR spectrum of SBP after adsorption showed that changing of the functional groups of PR was presented by the stretch vibration of amine N-H and aromatic C=C, stretching vibrations for azo compound N=N. The remarkable data attained that the stink bean peel can be used as an alternative biosorbent for the removal of organic pollutants including the textile effluent industry.

Keywords: Reactive dye; stink bean peel; kinetic studies; isotherm.

1. INTRODUCTION

Dyes are an organic compound that gave bright and lasting color to other substance and has been progressively utilized as a part of the material in textile, leather, paper, rubber, plastics, cosmetic industries, nourishment commercial enterprises as well as pharmaceuticals since these frequently have a complex aromatic bond which is more persistent. A lot of colors are commercially manufactured by industries, and parts of them are discarded into the environment during production and implementation. The disposal of dyes-contained wastewaters into the aquatic ecosystem without any treatment obstructed not only aesthetically unpleasant but also are elementary source for eutrophication and perturbations in aquatic life such as photosynthesis [1-7]. Numerous dyes present in the environment, hazards owing to their toxicity. The most common synthetic dye was utilized in the textile industry is an azo class which extensively becomes a major pollutant in dye effluents. Azo dyes consisting azo formation (-N=N-) bonded with other phenolic groups which have been reported that some of them are toxic, mutagenic and carcinogenic compounds majority of azo dyes have half-life greater than 2000h under daylight and imperviousness to biological even chemical degradation which harmfull to living organism even at scant concentration [7-9].

The need to develop more effective and economical treatment cost which consumes less chemical and energy recently

is significant for most textile factories which produce very large water pollution. A conventional method such as biological treatment even though low cost but less persuasive to remove toxic compounds such as dye [9-11]. Adsorption onto agriculture waste has been promising to be one of the reliable physicochemical treatment methods to remove dye such as pineapple leaf peel, lime peel, almond shell, and other leaf-based adsorbents [7, 12, 13].

Parkia speciosa (stink bean) is the most widespread species of the plant family Fabaceae which are prominent in Indonesia, Malaysia, Thailand, Philippine and other south Asian countries. The plant produce bears long fruits which consist of flat edible beans pods with bright green seeds. The seeds are typically isolated from the peels before use and normally applied in the medical industry due to restorative property, for example, anticancer. However, the peel was regarded as waste material and generally have not much attention in perspective to be recycled or reused instead of released [14-16].

The study aimed to examine the SBP as a biomass-based adsorbent to adsorb reactive azo dye PR. Batch adsorption studies were conducted by using varied conditions such as dosage, adsorption time, and initial concentration. The physical and chemical characteristic of adsorbent, kinetic and equilibrium studies were also determined in this study.

2. MATERIALS AND METHODS

2.1. Dye solution and adsorbent.

Procion Red Mx-5B was obtained from Sigma-Aldrich with high laboratory grade. The physicochemical characteristics of

the dye are shown in Table 1. The stock solution was made by dissolving 1 g of PR in 1 liter of distilled water. The series of 100, 200, 400, 600 and 800 mg/L of PR concentration was provided

from this main dye solution. The stink bean was collected from a local market and cleaned by tap water to remove the dirt. The SBP was ground and dried in the oven at 105°C for 24h, and sieved through 600 µm siever to obtain a similar average size of the adsorbent powder and stored it in an airtight container at room temperature.

2.2. SEM and FTIR.

The JEOL 6335F-Scanning Electron Microscopy (SEM) was used to analyse the distribution of pore and pore size. The role of functional groups involved in theadsorption process was analysed by a Spectrum One Perkin Elmer Fourier transform infrared spectrometry (FTIR) with frequency from 400 to 4000 cm⁻¹.

2.3. Equilibrium study.

Adsorption experiment was conducted in 100-ml Erlenmeyer flasks containing 50 ml solution and dried powder of SBP (5 g). The flask was sealed and agitated using a mechanical shaker (100 rpm) at room temperature (±27°C). The kinetics was studied in the duration of up to 24 hr. The effect of PR concentration was observed by the addition of PR from 100 to 1000 mg/L. The adsorbent loading was observed by the addition of SBP from 20 to 140 mg/L. The sample solution was filtered by using The Advantec filter paper and the removal of PR was measured by calculating the decrease of absorbance at 538 nm on the UV spectrum (NANOCOLOR VIS, Macherey-Nagel, Germany). The PR removal (%) and adsorption capacity qe (mg/g), was calculated by:

Removal (%) =
$$\frac{C_o - C_t}{C_o} \times 100$$
 (1)

Adsorption capacity =
$$\frac{C_o - C_t}{m} \times v$$
 (2)

Where Co and Ct are the liquid-phase concentrations of PR at initial and equilibrium (mg/L), v is the volume of PR solution (L), and m is the mass of adsorbent (g) [7, 17].

Table 1. Physical and chemical characteristics of PR.			
Properties	Value		
Structure	0,0		
	NaO S ONa		
	N ^z N		
	CI_N_NH OH		
	N N		
	CI		
Molecular Formula	$C_{19}H_{10}CI_2N_6Na_2O_7S_2$		
Appearance	Red powder		
CAS Number	12226-38-9		
Molecular weight	615.33 g/mol		
Maximum wavelength (λ_{max})	538 nm		

3. RESULTS

3.1. Characteristics of adsorbents.

The characteristic of adsorbent surface, pore size, and pore distribution is an important factor to evaluate the behavior of the functional group of the adsorbent. Typically, agricultural biomass has a similar chemical structure that playing an important role in bind adsorbate [17]. FTIR spectrum of SBP during the adsorption process is shown in Fig. 1. Many adsorption peaks occurred on the infrared spectrum. The functional group and corresponding infrared adsorption frequency of PR were reported in Table 2. The stretch vibration of broadband around 3367 cm⁻¹ indicated the occurrence of the hydroxyl bond. The involvement of methyl (-CH₃) in the adsorption process was observed at 2929 cm⁻¹ by asymmetrical stretch vibration. The stretch vibration of C=O bond was observed at 1620 cm⁻¹, and can be concluded as the involvement of carboxylic acid with intermolecular hydrogen bond during the adsorption process [18]. The involvement of methylene group and silicon oxide was indicated by the symmetric bending of -CH₃ at 1445 cm⁻¹, asymmetrical bending vibration of -CH₃ and -CH₂ at 1375 cm⁻¹, and stretching and bending of Si-O at 1230 cm⁻¹. The peak at 1035 cm⁻¹ was assigned to the stretching vibration of C-O-C and the bending vibration of -OH in the lignin component of the SBP [19, 20]. The FTIR spectrum of SBP after adsorption showed that most of the functional groups of PR appeared after treatment. The presence of stretching vibration of amine N-H and aromatic C=C was indicated by the peak at 3409

cm⁻¹, 2925 cm⁻¹, and 1449 cm⁻¹. The stretching vibrations at 1671 cm⁻¹ had indicated the occurrence of azo compound N=N. The peak at 1375 and 1324 cm⁻¹ was assigned to the involvement of amine bond C-N in the adsorption process. Finally, the presence of sulfones group S=O halogen group was represented by the peak at 1104 cm⁻¹ and 613 cm⁻¹.

Table 2. Functional group of SBP before and after adsorption.

Frequency (cm ⁻¹)		Assignment	
Before	After adsorption		
adsorption			
3367	3409	Bonded O-H groups	
2929	2925	CH ₃ groups	
1620	1620	C=O stretching	
1445	1449	CH ₃ groups	
1357	1374	CH ₃ , CH ₂ groups	
1230	1233	Si-O groups	
1035	1031	C-O-C groups	

The morphology of SBP structure before and after the adsorption was shown in Fig. 2. The porous texture obviously appeared with various pore sizes on the SBP surface. However, the structure of SBP was significantly changed after the adsorption. The raw SBP appears to have a rough pore on the surface which partially covered by PR molecules.

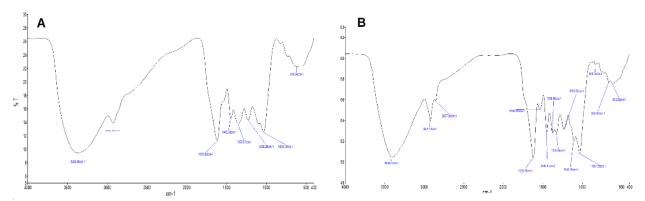


Figure 1. FTIR spectrum of SBP before (A) and after adsorption (B).

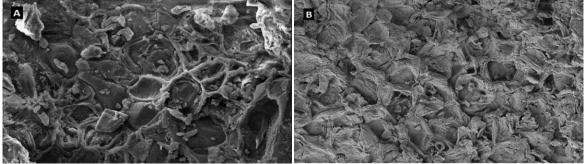


Figure 2. Morphology of SBP structure before (A) and after the adsorption (B).

3.2. Effect of some parameters.

The removal of PR was rapidly occurred in the initial 3 h due to the accessibility of dye compound to the active site of adsorbent, and gradually slower removal when reached the equilibrium at 12–24 h). The adsorbent surface was fully occupied with PR during equilibrium at removal maximum of 77.43%. The highest adsorption capacity (7.743 mg/g) was reached after 12 h to saturation of active pore site during removal. Furthermore, the adsorption phenomena of SBP take a relatively long contact time. For the most part the adsorbate, molecules were initially introduced adsorbent surface until the limit layer impact, then adsorb into the pore through diffusion into the permeable structure of the adsorbent [6, 19].

The effect of the dosage, adsorption time, and initial concentration is shown in Figure 3. The initial concentration of PR was set from 50 to 1000 mg/L. There is no significant removal was occurred during this treatment. The removal of PR was slightly reduced from 99.8 to 82.04 % when increasing the initial concentration. On the contrary, the adsorption capacity was improved from 0.499 to 8.204 mg/g when the initial concentration increased. The adsorption capacity was improved when the dye concentration raised because of the driving force of mass transfer as well as increase the potential of connection among the adsorbent and adsorbate [21, 22]. The rise of dosage leads to an increase in the removal of PR. The incremental of PR removal becomes very low at adsorbent concentration 100 mg/L and SBP surface tends to saturated at adsorbent concentration 120 mg/L.

The bigger surface area and availability of active sites occurred when increased the adsorbent dosage. The adsorption process becomes very low at adsorbent concentration 120 mg/L due to the surface of SBP and PR solution reach the equilibrium. The highest removal was reached at adsorbent dosage of 120

mg/L. On the contrary, the lowest adsorption capacity (7.52 mg/g) was observed at that dosage. Thus the effective adsorbent dosage for removal of PR from 50 mL aliquot of 1000 mg/L was 100 mg/L. A similar trend has been reported by previous studies [23, 24].

3.3. Isotherm and kinetic.

Adsorption isotherm which represented by invaluable curves describing the phenomena prevailing the release (retention) or movement of the substance in the watery permeable aquatic environment to a solid adsorption mechanism. The isotherm described the circulation of the substance molecule among the solid phase and the liquid phase was at an equilibrium level. Generally, mathematical correlation depicted by graph express together with physicochemical parameters with fundamental thermodynamic assumptions offers to understand for the relationship between active site of adsorbent and the adsorption mechanism [25, 26]. The Langmuir empirical model represents the adsorption of molecule in monolayer and the maximum adsorption capacity was determined by the imperative part. This model implicated that adsorption occurs at particular heterogeneous sites within the adsorbent which neglect all kind of interaction between adsorbed substance. Furthermore, the energy of adsorption considered constant and no dye molecule movement on the surface of adsorbent [4]. The Langmuir equation model can be expressed

Langmuir equation,
$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m}$$
 (3)

where C_e (mg/L) is the concentration of PR at equilibrium, q_e (mg/g) is the adsorption capacity at equilibrium, q_m is the maximum adsorption capacity corresponding to complete monolayer coverage on the surface (mg/g), K_L is the Langmuir constant which relates to the energy of adsorption (L/mg).

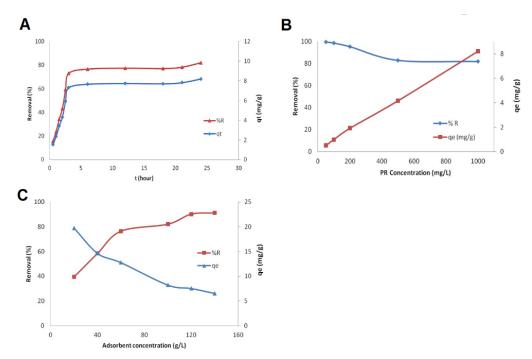


Figure 3. Effect of adsorption time (A), initial concentration (B) and dosage (C) on removal of PR by SBP.

Adsorption isotherm of PR has been observed by varied the amount of adsorbent dosage (1, 3 and 5 g). The plot of C_e/q_e versus C_e in Fig. 4A indicated the adsorption of PR by SBP performed a poor correlation coefficient (R^2) for Langmuir isotherm with R^2 = 0.6938, 0.6904 and 0.8433 for adsorbent dosage 1, 3 and 5g respectively. Value of q_m was achieved from the slope while K_L calculated from the intercept of a linear plot. Currently, the use of Freundlich isotherm model is intensively applied in heterogeneous surface energy system especially for high interactive species of activated carbon and molecular sieve. This model is mostly implemented for more than one-layer adsorption (multilayer) with a non-uniform circulation of adsorption heat and affinities over the heterogeneous surface. The Freundlich isotherm can be represented as:

Freundlich equation,

$$Inq_e = InK_F + (\frac{1}{n})InC_e \tag{4}$$

In general, K_F value rises when the capacity of adsorbent increases. The value of 1/n, indicated the favorability of adsorption. It's represented the quantity of adsorption strength of surface heterogeneity. Typically, the values of n > 1 more favorable adsorption condition and 1/n closer to zero specified the more heterogeneous adsorption [4, 7, 25, 27]. The linear plot in Fig. 4B shows that adsorption of PR by SBP has a good fitting curve for Freundlich isotherm with the $R^2 = 0.9993$, 0.9786 and 0.9728 for 1, 3 and 5 g of adsorbent dosage. The values of K_F and n can be calculated from the intercept and the slope of the graph as stated in Table 3.

The Temkin isotherm contains an element that unequivocally considers the adsorbent-adsorbate interactions with the effect of adsorption heat and the distribution of binding energies. The Temkin constants, B (Jmol⁻¹) which related to

adsorption heat and determined from the slope of adsorption capacity versus ln Ce while equilibrium binding constant, A (L/g) associating with the extreme binding energy was calculated from the intercept of the plotting graph. The equation of Temkin isotherm is as follows:

Temkin equation,
$$q_e = BInA + BInC_e$$
 (5)

Linear plot of q_e versus Ln C_e for the sample of PR Fig. 4C shows that adsorption does not follow the Temkin model which $R^2 = 0.7869$, 0.7714 and 0.7482 for adsorbent dosage 1, 3 and 5 g respectively. Hence, by comparing the correlation factor, Freundlich was the most appropriate isotherm model to represent the PR removal by SBP. The result suggested that adsorption is favourable with values of n > 1 for all adsorbent amount. The intensity value increased in a high amount of adsorbent dosage indicated the adsorption process was more favourable with n = 1.67, 1.68 and 2.80 for 1, 3 and 5 g respectively. A similar observation has been reported by in removal of synthetic dye by adsorbent prepared from raw biomass and activated carbon. Table 3 specified the K_F value of SBP was low compared to other adsorbents. The PR sorption kinetic was calculated by determining the effect of contact time on the PR adsorption for 24 h. As illustrated in Fig. 3, kinetics of the adsorption of PR was achieved equilibrium at 12 h. The significant to evaluate the reaction kinetic is to know the uptake mechanism of dye by the adsorbents in terms of the order of rate constant. Therefore, the adsorption of PR by SBP could be evaluated by specifying the kinetic model such as Pseudo-first-order, Pseudo-second-order and intraparticle:

Pseudo-first-order, In
$$(q_e - q_t) = \text{In } q_e - k_l t$$
 (6)

Pseudo-second-order,
$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$
 (7)

Intraparticle diffusion, $qt = k_{diff} t^{\frac{1}{2}} + C$ (8)

Where q_e and q_t (mg/g) respectively refer to the amount of PR adsorbed at equilibrium and at time t (min); k_1 is the rate constant of pseudo-first-order, k_2 is rate constant of pseudo-second-order; k_{dif} is the intraparticle diffusion rate constant (mg/g min^{1/2}), and C is the intercept which indicates the boundary layer thickness. The larger the intercept is the greatest boundary layer effect.

Table 3. Freundlich Isotherm for adsorption of dyes by the various adsorbent.

Adsorbate	Adsorbent	$\mathbf{K}_{\mathbf{F}}$	References
Basic dye	Granular activated carbon	77.82	[28]
Basic dye	Natural zeolite	2.574	[28]
Acid Red 57	Acid activated bentonite	3.18	[29]
Congo Red	Coir pith activated carbon	2.53	[30]
Effluent	Mahogany sawdust	88.3	[31]
wastewater's			
dye			
Procion Red	Stinky Bean Peel	0.994	This study

The value of rate constant (k_1) for the adsorption of PR by SBP can be obtained from the slope of $\ln(q_e-q_t)$ against t while rate constant (k_2) can be calculated from the plot of t/q_t versus t and k_{diff} can be determined from q_t versus $t^{1/2}$. Table 4 represented the kinetics parameters of PR adsorption. The results show that the pseudo-second-order model was the most appropriate model to

represent the kinetic model as shown by the correlation coefficient of 0.995. Furthermore, the value of experimental q_e ($q_{e,exp}$) was much closer to the calculated value of qe ($q_{e,cal}$) which were 8.204 (mg/g) and 8.69 (mg/g) respectively for the pseudo- second-order model. The adsorption process controlled by the particle diffusion if the good fitting curve performed by the plot of q_t versus $t^{1/2}$. Table 4 indicated the poor correlation coefficient (0.5796) for the diffusion mechanism of PR into SBP. The result gives an idea the diffusion process does not contribute to intra-particle diffusion.

Table 4. Kinetic parameter of PR adsorption

Kinetic model	Parameter
q _e experiment (mg/g)	8.2040
Pseudo-first-order	
q _e calculated (mg/g)	1.8123
K_{f}	0.0007
$R^{\frac{1}{2}}$	0.6587
Pseudo-second-order	
qe calculated (mg/g)	8.6881
K _s	0.0010
\mathbb{R}^2	0.9950
Interparticle- diffusion	
I	3.7544
$\mathbf{k_{id}}$	0.0064
R^2	0.5796

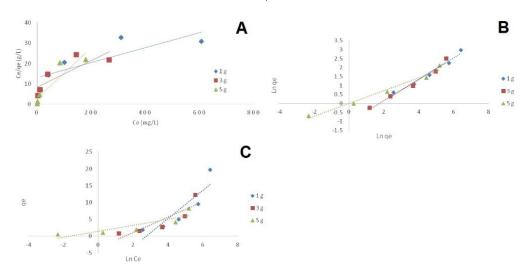


Figure 4. Langmuir (A), Freundlich (B), and Temkin (C) isotherm model for the adsorption of PR by SBP.

4. CONCLUSIONS

The present study proved that the raw SBP can be used as an alternative biosorbent for the removal of varied organic pollutants including the textile dye. The adsorption equilibrium (77.43%) was reached after 12 h of contact time. The removal of PR was slightly reduced from 99.8 to 82.04 % when increasing the

initial concentration. The rise of dosage leads to an increase in the removal of PR. The Freundlich ($R^2 = 0.9993$) and pseudo-second-order ($R^2 = 0.995$) was the most appropriate isotherm model and kinetic model to represent the PR removal by SBP.

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