



Adsorption of methylene blue from aqueous solution by peanut shell based activated carbon

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ABSTRACT

Peanut shell-based activated carbon (PSAC) was used to study the removal of methylene blue (MB) dye from aqueous solution. Chemical agent of sodium hydroxide (NaOH) and gasification agent of carbon dioxide (CO₂) were used to prepare PSAC via physicochemical activation. In batch studies, several effects namely, initial MB dye concentration, solution temperature, and solution pH were evaluated. Isotherm and kinetic study revealed that the adsorption system of MB-PSAC was fitted the finest by Freundlich and pseudo-second-order models, respectively. Thermodynamic studies certified that MB adsorption onto PSAC was physically controlled and was endothermic in nature.

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1. Introduction

The demand for dyes to colour the products especially in textile industry is kept increasing with the increasing of world's population. Although this is good to boost the economy, the discharge of wastewater containing dyes without a proper treatment is causing severe problems to the receiving water bodies. Besides textile industry, dyes wastewater are also coming from other industries like paint, fibre, tannery, plastic and battery manufacturing industry [1]. Dyes are known to be non-biodegradable, imparts toxicity into environment and causing damage to aquatic life [1]. Furthermore, most dyes are having carcinogenic, mutagenic and endocrine disruptor properties which is harmful to humans and animals [2]. Methylene blue (MB) is an example of azo dye that is extensively used in textile industry. Due to the existence of azo group in its chemical structure, MB dye is extremely stable towards chemical and biological degradation.

Scientists around the world are working hard to develop many technologies to treat wastewater containing dyes, namely membrane filtration, electro-coagulation, ion exchange, advanced oxi-

dation process and activated sludge. However, none of these technologies can hold a candle to adsorption process, a process which can be defined as a surface accumulation of a specific compound which occurs at interface of the two phases [3]. Main advantages of adsorption process include: (i) relatively simple in operation and design, (ii) able to remove pollutants even at a very low concentration, (iii) can be carried out in batch and continuous processes, (iv) resistance towards toxic substances, (v) relatively low cost and (vi) available for regeneration and reuse [4]. Nonetheless, adsorption process via activated carbon (AC) as an adsorbent is proven to be versatile in removing variety of pollutants such as dyes, heavy metals, bisphenol A, carbon dioxide, CO₂, chlorophenols and many others.

Despite of all its advantages, commercially available AC is still considered as expensive because it is derived from non-renewable precursor such as coal. Because of that, researchers nowadays are exploring new precursor especially agricultural wastes to be converted into AC [5]. In this work, peanut shell is selected to be the precursor for AC production to treat MB dye in aqueous solution. Peanut shell is relatively hard and dry physically, which makes it a good precursor for AC. Peanut (*Arachis hypogaea*) is one of the main food crop that is grown commonly in Asia, Oceania, Africa and America, with global production up to 46.6 million tons in

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the year 2017–2018 [6]. For every kg of peanut, peanut shell with the amount of 230–300 g are generated and they end up to be a waste due to their limitation of use [7]. In Malaysia, peanuts are popular to be eaten raw or processed into peanut butter or to be used in cooking. Most peanuts in Malaysia are imported from other countries such as Indonesia, China and India. In literature, some researchers had successfully converted peanut shell into AC to adsorb MB dye, acid yellow 36 dye, hydrogen sulfide, H_2S , direct blue-86 dye, Cr (VI) and Pb (II). However, there is none of work reported in the literature about the conversion of peanut shell into AC using sodium hydroxide, NaOH coupling with carbon dioxide, CO_2 as activating agents.

2. Experimental

2.1. Materials

Peanut shell (PS) was collected from the Thong Thye Factory Sdn. Bhd., Sungai Siput, Perak, Malaysia. 0.050 M hydrochloric acid (37% purity) was used to alter the pH of the dye solution and sodium hydroxide, NaOH in pellet form (99% purity) was used as a chemical activating agent. Both of these chemicals were obtained from Mallinckrodt (USA). Properties of MB dye obtained by Sigma Aldrich (M) Sdn. Bhd, Malaysia is shown in Table 1. Nitrogen, N_2 and carbon dioxide, CO_2 were used for the carbonization and activation processes, respectively.

2.2. Preparation of peanut shell based activated carbon

The PS was first cleansed with water and dried overnight at temperature of 105 °C in an oven. Carbonization process was performed in a stainless steel vertical tubular reactor. Dried peanut shell was placed on top of a metal mesh inside the reactor. The temperature of the furnace was set to increase from room temperature to 500 °C under a purified N_2 flow at 150 mL/min to produce char. Carbonization step was carried out for 2 h. The resulted char was then impregnated with NaOH using mass ratio of 1:1. Specific amount of char and NaOH pellet were mixed together with deionized water to dissolve the NaOH powder. The mixture was kept inside oven for 24 h at temperature of 105 °C to allow dehydration process to occur. Next, activation step was carried out. NaOH-impregnated char was pyrolyzed starting from room temperature to 800 °C under N_2 gas. Once the temperature achieved 800 °C, the flow of N_2 was changed to CO_2 and prolonged for 2 h. After that, the activated sample was allowed to cool to room temperature under the flow of N_2 gas. The activated sample was then undergo washing step with hot deionized water to remove excess NaOH on it. The washing process was repeated for several time to ensure the pH of washing solution became 7. The wet PSAC was then dried in an oven overnight.

2.3. Characterization system

Volumetric adsorption analyzer (model: Micromeritics ASAP 2020) was used to determine: (i) surface area, (ii) pore volume

Table 1
Properties of MB dye.

Dye name	Methylene Blue
Molecular formula	$C_{16}H_{18}N_3SCl$
Molecular weight, g/mol	319.850
CAS no.	61-73-4
λ max. (nm)	661
Chemical structure	

and (iii) average pore diameter of the samples. The equation of Brunauer-Emmet-Teller isotherm was used to compute the BET surface area of the samples. An estimation was made for the total pore volume to be the liquid volume of nitrogen at a relative pressure of 0.980. Scanning electron microscope, SEM (model: Quanta 450 FEG, Netherland) was used to examine surface morphology of the samples. The proximate analysis was carried out by using simultaneous thermal analyzer (model: Perkin Elmer STA 6000, USA). The surface characteristic of the samples were characterized using a FTIR spectrometer (model: IR Prestige 21 Shimadzu, Japan).

2.4. Equilibrium and kinetic study

The experiment was performed to see how different conditions (initial MB concentration, solution temperature and solution pH) were affecting the adsorption performance of MB onto PSAC. A stock solution of MB dye (1000 mg/L) was prepared in a volumetric flask by mixing 1 g of MB dye powder in 1000 mL of deionized water provided by USF ELGA. The solution was withdrawn to determine the concentration by UV-Visible spectrophotometer. Eq. (1) and Eq. (2) were used to calculate the amount of MB dye adsorbed at equilibrium, q_e (mg/g) and percent removal of MB dye, respectively:

$$q_e = \frac{(C_o - C_e)V}{W} \quad (1)$$

$$\% \text{Removal} = \frac{(C_o - C_e)}{C_o} \times 100\% \quad (2)$$

C_o and C_e (mg/L) are the concentration of MB dye at initial and at equilibrium, respectively. V and W are the volume of MB dye solution and mass of PSAC used, respectively. The data obtained were then fitted into Langmuir, Freundlich, Temkin, Dudinin-Radushkevich, Koble-Corrigan, Radke-Prausnitz, Vieth-Sladek and Brouers-Sotolongo models, respectively. For kinetic studies, the amount of adsorbed MB dye at time q_t was calculated by:

$$q_t = \frac{(C_o - C_t)V}{W} \quad (3)$$

For kinetic study, data was applied into non-linear pseudo-first-order (PFO), non-linear pseudo-second-order (PSO), Elovich and Avrami models [8]. The value of correlation coefficient, R^2 was compared to determine the best isotherm model and kinetic model to describe MB-PSAC adsorption system

3. Results and discussion

3.1. Characterization of samples

PSAC was found to have BET surface area of 868.750 m^2/g , total pore volume of 0.459 m^3/g and pore diameter of 3.760 nm respectively, as shown in Table 2. Relatively high of BET surface area together with pore volume were a manifestation of the effectiveness of physicochemical activation employed in this study. During chemical activation, the metallic sodium was free to move to penetrate deep into sample's skeleton, therefore creating pores. Then, during physical activation, CO_2 molecules move vigorously and bombarded the PSAC's skeleton, thus creating more pores.

The moisture content, volatile matter and ash were very high in PS but low in PSAC as shown in Table 3. Heat treatment during carbonization had caused the moisture and volatile matter to vaporize and leaving the sample. Fixed carbon that was relatively high and moisture content that was relatively low were an indication of a good adsorbent.

Fig. 1 shows the SEM micrographs of PS and PSAC samples. The surface of PS was found to be absent from any pores. After

Table 2

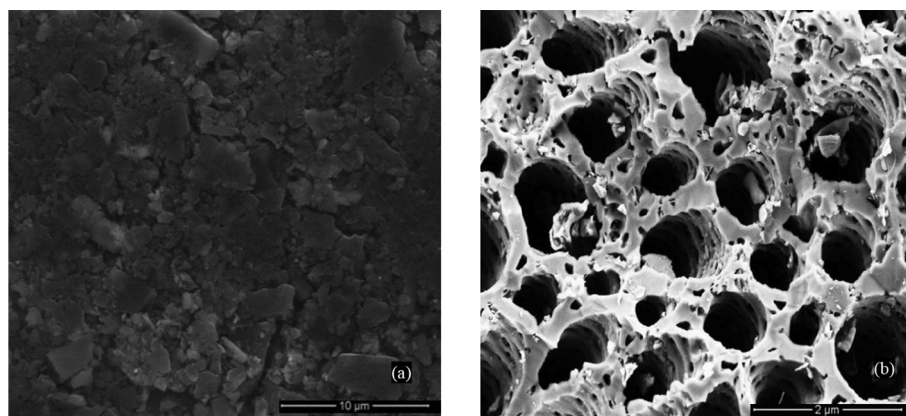
Surface area and pore characteristics of the samples.

Sample	BET surface area (m ² /g)	Mesopores surface area (m ² /g)	Total pore volume (cm ³ /g)	Average pore diameter (nm)
PS char	175.380	96.800	0.131	3.520
PSAC	868.750	474.220	0.459	3.760

Table 3

Proximate analysis.

Sample	Proximate analysis (%)			
	Moisture	Volatile matter	Fixed carbon	Ash
PS	6.250	55.740	30.690	7.320
PS char	3.600	29.640	61.330	5.430
PSAC	2.840	17.650	74.730	4.780

**Fig. 1.** (a) SEM image of PS ($\times 10k$); (b) SEM image of PSAC ($\times 50k$).

activation treatment, several pores formed on PSAC surface. The pores development were due to the activation process via NaOH and CO₂ as activating agent.

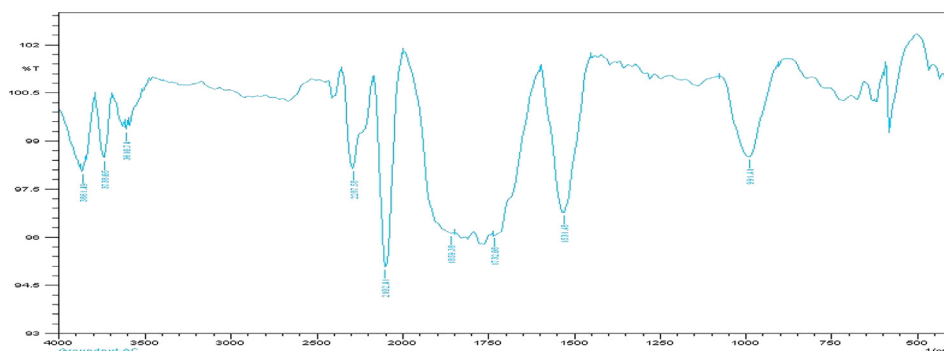
Peanut shell consists of various functional groups on its surface as shown in Fig. 2. O–H stretching bonds were detected at bandwidth around 3300–3700 cm⁻¹ which indicates the existence of hydroxyl functional groups comprising hydrogen bonding. Other major peaks recognized at range of 1000–1300 cm⁻¹ and 1500–2100 cm⁻¹ shows the S=O and C=O stretching, respectively.

At bandwidth of 500–750 cm⁻¹, the peaks present could be assigned to C–H out-of-plane bending in benzene derivatives and was found to reduce in PSAC. This indicates the functional groups disappeared due to heat instability during activation process where bonding was broken due to high temperature. Thus, leading to destruction of functional groups, either partially or completely.

3.2. Batch adsorption equilibrium studies

Fig. 3(a) shows the adsorption uptake whereas Fig. 3(b) shows the percentage of MB removal from the solution at 30 °C. The amount of MB dye adsorbed, q_t onto PSAC increased with time until finally reaches its equilibrium where PSAC could not adsorb any MB molecules. At initial stage, more available surface sites for MB molecules to be adsorbed onto PSAC. Large amount of available sites on PSAC provided a greater amount of driving force for MB molecules to defeat the barrier of mass transfer resistance between solid phase and liquid phase.

After sometimes, it was getting tougher for the available sites to be occupied because repulsive force was formed between MB molecules on the surface of PSAC and MB molecules in bulk phase [9]. Longer contact time was required for higher initial concentra-

**Fig. 2.** Surface chemistry of PSAC.

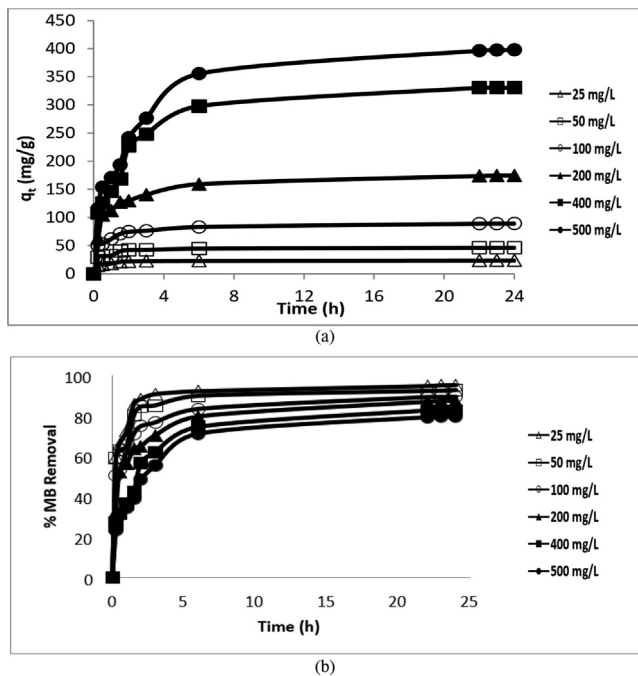


Fig. 3. (a) MB adsorption uptakes versus time and (b) MB dye removal percentage versus time at various initial concentration.

tion in order to achieve equilibrium state. Lower initial concentration of 25 mg/L required 6 h to attain equilibrium state while higher initial concentration of 500 mg/L needed 20 h to reach the same state.

MB removal was found to be 412.312, 422.765 and 434.268 mg/g for solution temperature of 30, 45 and 60 °C, respectively. An increase in MB removal with temperature indicates an endothermic behaviour of an adsorption system. At elevated temperature, an extra energy was supplied to MB dye molecules to react with active sites that available on the surface of PSAC. Furthermore, rate

of diffusion also increased at higher temperature, therefore more MB dye molecules were able to cross the external boundary layer and entered the internal pores of PSAC [10].

The MB removal was increased from 75.320% to 92.450% as the pH of the solution increased from 2 to 8, respectively. This was due to the chemical structure of MB dye where it undergoes protonation in the acidic medium. At lower pH, number of positive ion increases which does not favours the MB cation removal where it has to compete with excess H^+ ion for the adsorption site on PSAC. In the range of pH 8 to 12, the MB removal was almost constant.

3.3. Adsorption isotherms

The constants and R^2 values for each equations were tabulated in Table 4. For MB-PSAC adsorption system, the isotherm models fitted the data the best, in the following order: Radke-Prausnitz > Vieth-Sladek > Brouers-Sotolongo > Freundlich > Koble-Corrigan > Langmuir > Temkin > Dubinin-Radushkevich.

The separation factor R_L in Langmuir isotherm is a useful parameter which reflects the favourability of the adsorbate-adsorbent adsorption system. The values of R_L were found to be 0.656, 0.488, 0.323, 0.192, 0.106 and 0.087 for initial dye concentration of 25, 50, 100, 200, 400 and 500 mg/L, respectively, at 30 °C. These values were in the range from 0 to 1, thus signified that the MB-PSAC adsorption system was favourable.

3.4. Adsorption kinetic

Adsorption process that took place in an adsorbate-adsorbent system is a time dependant process. Therefore, the involvement of kinetics model is a must to identify the reaction rate and reaction mechanism. Kinetic models are also useful in providing information such as the residence time needed to complete the adsorption process. The values for constant in each kinetic models at 30 °C is shown in Table 5. The kinetic models fit in MB adsorption onto PSAC are in following sequence: pseudo-second-order > pseudo-first-order > Avrami > Elovich.

Table 4
Isotherm parameters for MB at 30 °C.

Langmuir	Freundlich	Temkin	Dubinin-Radushkevich	Koble-Corrigan	Radke-Prausnitz	Vieth-Sladek	Brouers-Sotolongo
$q_m = 555.600$	$K_F = 3.710$	$B = 85.150$	$q_s = 175.300$	$A_{KC} = 21.320$	$Q_m = 540.800$	$k_{VS} = 0.500$	$Q_m = 560.200$
$K_L = 0.020$	$n_F = 1.540$	$A = 0.560$	$E = 745.400$	$B_{KC} = 0.050$	$k_{RP} = 0.020$	$Q_m = 539.800$	$k_{BS} = 0.670$
			$B_{DR} = 9 \times 10^{-7}$	$n_{KC} = 1.000$	$m_{RP} = 0.960$	$\beta_{VS} = 0.020$	$\alpha_{BS} = 0.020$
$R^2 = 0.936$	$R^2 = 0.978$	$R^2 = 0.889$	$R^2 = 0.598$	$R^2 = 0.971$	$R^2 = 0.989$	$R^2 = 0.939$	$R^2 = 0.919$

Table 5
Constant parameters of kinetic for adsorption of MB at 30 °C.

Model	Kinetic parameters	Initial MB dye concentration (mg/L)					
		25	50	100	200	400	500
Experimental data	q_e (mg g ⁻¹)	23.081	46.293	89.282	174.470	330.383	397.584
Pseudo-first order	k_1 (min ⁻¹)	0.381	0.363	0.347	0.334	0.327	0.311
	q_e (mg g ⁻¹)	20.625	42.854	81.785	163.857	319.635	378.865
	R^2	0.991	0.923	0.945	0.988	0.993	0.968
Pseudo-second order	k_2 (min ⁻¹)	0.321	0.317	0.309	0.302	0.295	0.291
	q_e (mg g ⁻¹)	23.058	46.114	90.004	173.245	330.895	395.754
	R^2	0.999	0.999	0.999	0.999	0.999	0.999
Elovich	α (mg g ⁻¹ min ⁻¹)	61993.000	58380.961	13347.277	3736.808	1316.135	1253.147
	β (g mg ⁻¹)	0.557	0.274	0.114	0.052	0.025	0.022
	R^2	0.824	0.858	0.949	0.947	0.955	0.968
Avrami	k_{AV} (min ⁻¹)	0.064	0.051	0.032	0.021	0.017	0.014
	n_{AV}	0.423	0.415	0.482	0.584	0.721	0.680
	R^2	0.963	0.964	0.976	0.956	0.960	0.968

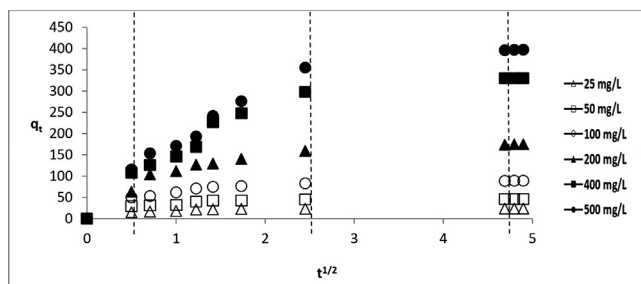


Fig. 4. Plots of intraparticle diffusion model for MB dye adsorption onto PSAC.

Fig. 4 shows the intraparticle diffusion plots for MB removal at 30 °C for different dye initial concentration. The first sharper region in the plot, which took about 0.5 h is the instantaneous adsorption, which is likely caused by powerful electrostatic attraction between adsorbates and adsorbent. The second stage that occurred from 0.5 h to 2.5 h is known as gradual adsorption stage, which associated with intraparticle diffusion [11]. On the other hand, the third stage from 2.5 h until equilibrium point is a stage where the slowing down of intraparticle diffusion starts to take effect because the adsorbate concentration in the solution is getting low [12]. The plots were non-linear over the entire time range which could be explained by the fact that besides intraparticle diffusion, other type of mechanism was also involved as rate limiting step.

The values of constant for intraparticle diffusion model are presented in Table 6. The value of k_{diff} was found to increase when MB dye initial concentration increased. This could be explained by the fact that at higher initial concentration, driving force is greater, therefore, enhancing dye diffusion rate [13]. Higher value of C at higher initial concentration signified the increased of boundary layer thickness [14].

Boyd plot was used to further investigate the actual slowest step in MB-PSAC adsorption system, as shown in Fig. 5. Since all plots did not go through origin, therefore, it is confirmed that the adsorption process was governed by film type of diffusion.

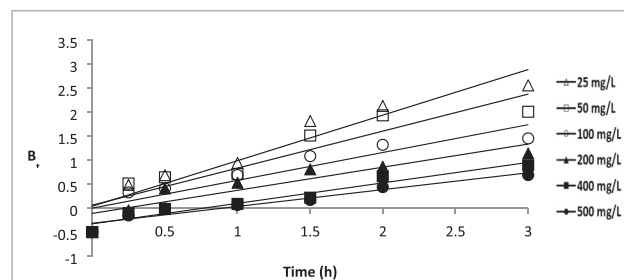
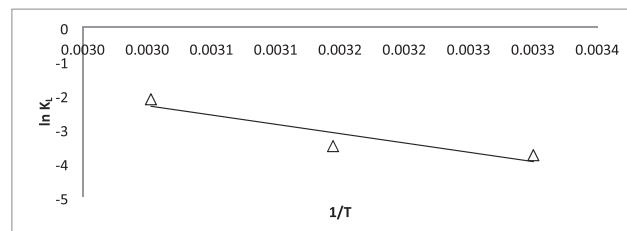
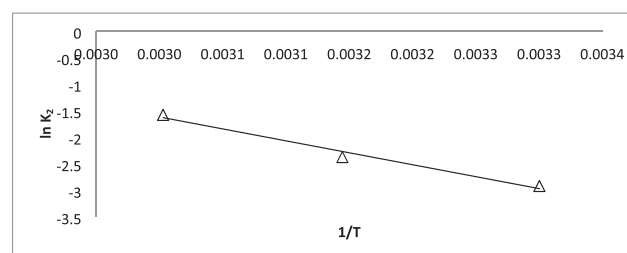


Fig. 5. Boyd plots for MB on PSAC at 30 °C.



(a)



(b)

Fig. 6. (a) Plots of $\ln K_1$ versus $1/T$ and (b) Plots of $\ln k_2$ versus $1/T$ for adsorption of MB on the PSAC.

3.5. Adsorption thermodynamic

Fig. 6(a) shows the plot for Van't Hoff equation for MB dye at different temperature. Fig. 6(b) shows the plot of k_2 versus $1/T$ where we could compute the values of activation energy from the slope from each plot. Each plot shows negative slope indicates the positive values in activation energy. Data for constants for thermodynamic studies (ΔH° , ΔS° , E_a and ΔG°) were computed and tabulated in Table 7.

The value of ΔH° was positive which reflects the endothermic behaviour of the MB-PSAC adsorption system. Endothermic adsorption was known to be caused by the increasing rate of diffusion of MB dye molecules into PSAC's available active sites when the solution temperature was increased. The positive value of ΔS° indicated that the randomness of the reaction occurred at the interface of solid-liquid face was increased. In addition, the ΔG° shows negative values which reflects the spontaneous behaviour

Table 7

Thermodynamic parameters for MB adsorption on PSAC.

ΔH° (kJ/mol)	ΔS° (J/mol)	E_a (kJ/mol)	ΔG° (kJ/mol)		
			303 K	318 K	333 K
45.473	1117.042	37.424	-35.418	-37.172	-38.931

of the adsorption process [15]. The negative values in MB removal shows the reaction rate increases with increasing in temperature [16]. Furthermore, E_a value was positive which show the feasibility of the adsorption process. The value for E_a was found to be near with 40 kJ/mol, therefore it can be deduced that the adsorption process was governed by physisorption [17]. Physical type of adsorption is generally faster compared to the chemical type of

Table 6

Intraparticle model constant and correlation coefficients for MB adsorption on PSAC at 30 °C.

Initial concentration (mg/L)	k_{diff1} (mg/g h ^{1/2})	k_{diff2} (mg/g h ^{1/2})	k_{diff3} (mg/g h ^{1/2})	C_1	C_2	C_3	R_1^2	R_2^2	R_3^2
25	29.764	3.681	0.614	0.000	15.422	20.854	1.000	0.712	0.878
50	58.868	7.993	1.083	0.000	27.573	40.993	1.000	0.761	0.949
100	100.281	16.369	0.881	0.000	46.862	84.918	1.000	0.859	0.996
200	126.784	31.832	3.860	0.000	83.519	155.711	1.000	0.976	0.775
400	215.337	104.909	0.746	0.000	53.002	326.730	1.000	0.942	0.923
500	231.249	122.161	6.425	0.000	58.254	366.174	1.000	0.981	0.954

adsorption and physical adsorption is usually occurs at low temperature.

4. Conclusion

In this study, peanut shell was successfully converted to PSAC to scavenge MB dye from aqueous solution. PSAC posed BET surface area and adsorption capacity of 868.750 m²/g and 555.600 mg/g, respectively. Batch studies found that more time was needed for higher initial concentration to attain equilibrium as compared to lower initial concentration that required shorter time to attain the same stage. Isotherm studies divulged that Freundlich model fitted the data the best whereas kinetic studies disclosed that pseudo-second-order fitted the data the finest. Thermodynamic studies revealed that MB-PSAC adsorption system was endothermic in nature, governed by physisorption, feasible and spontaneous.

CRedit authorship contribution statement

Mohd Azmier Ahmad: Writing - review & editing. **Mohamad Firdaus Mohamad Yusop:** Conceptualization. **Ridzuan Zakaria:** Writing - original draft, Investigation. **Jamilah Karim:** Data curation. **Nasehir Khan E.M. Yahaya:** Project administration. **Muhammad Azroie Mohamed Yusoff:** Formal analysis. **Noor Haza Fazlin Hashim:** Resources, Software, Supervision, Validation, Visualization. **Nor Salmi Abdullah:** Methodology.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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