



Evaluation of papaya seeds as a novel non-conventional low-cost adsorbent for removal of methylene blue

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ABSTRACT

The feasibility of using papaya seeds (PS), abundantly available waste in Malaysia, for the cationic dye (methylene blue) adsorption has been investigated. Batch adsorption studies were conducted to study the effects of contact time, initial concentration (50–360 mg/L), pH (3–10) and adsorbent dose (0.05–1.00 g) on the removal of methylene blue (MB) at temperature of 30 °C. The equilibrium data were analyzed by the Langmuir, the Freundlich and the Temkin isotherms. The data fitted well with the Langmuir model with a maximum adsorption capacity of 555.557 mg/g. The pseudo-second-order kinetics was the best for the adsorption of MB by PS with good correlation. The results demonstrated that the PS is very effective to remove methylene blue from aqueous solutions.

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1. Introduction

The textile dyeing industry consumes large quantities of water and produces large volumes of wastewater from different steps in the dyeing and finishing processes [1]. Effluent derived from the textile and dyestuff activities can provoke serious environmental impact in the neighboring receptor water bodies because of the presence of toxic reactive dyes, chlorolignin residues and dark coloration [2]. There are many treatment processes applied for the removal of dyes from wastewater. A critical review on current treatment technologies with a proposed alternative was reported by Robinson et al. [3].

Adsorption process using commercial-activated carbons is very effective for removal of dyes from wastewater, but its high cost has provoked the search for alternatives and low-cost adsorbents. Most of the adsorption studies have been focused on untreated plant wastes such as oil palm ash [4], wheat bran [5], rice husk [6], rice bran and wheat bran [7], oil palm trunk fibre [8], yellow passion fruit peel [9], sugarcane dust [10], durian (*Durio zibethinus* Murray) peel [11], guava (*Psidium guajava*) leaf powder [12], almond shells [13], coffee husks [14], broad bean peels [15], pomelo (*Citrus grandis*) peel [16] and granular kohlrabi peel [17]. Recently, an extensive list of sorbent literature for dye removal has been compiled by Allen and Koumanova [18].

In this work, an attempt to use papaya (*Carica papaya* L.) seeds, as a novel non-conventional low-cost adsorbent for removal of methylene blue (MB) from aqueous solution has been made. Papaya (*C. papaya* L.) is a tropical and subtropical crop. Malaysia is now capable of producing up to 72,000 tonnes of papaya annually. Papaya fruits produce solid waste such as peel/skin and seeds. The discarded portion of seeds can be between 15 and 20% of its weight. Due to the high consumption of papaya fruit, massive amounts of the seeds are disposed, causing a severe problem in the community. To make better use of this waste, it is proposed to use it as adsorbent to remove MB from aqueous solutions. Thus, the purpose of the present work was to evaluate the adsorption potential of papaya seeds (PS) for the removal of methylene blue from aqueous solution.

2. Materials and methods

2.1. Adsorbate

The methylene blue used in this work was purchased from Sigma–Aldrich. The MB was chosen in this study because of its known strong adsorption onto solids. MB has a molecular weight of 373.90 g/mol. The maximum wavelength of this dye is 668 nm.

2.2. Preparation of papaya seeds adsorbent

Papaya seeds used in this work were removed manually from a fresh papaya fruit obtained from the fruits market, Nibong Tebal, Penang, Malaysia. The seeds were washed with distilled water,

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boiled with water for 20 min, filtered out and dried in an oven at 60 °C for 24 h. The dried materials were crushed and sieved to desired mesh size (125–250 μm). The prepared PS sample was stored in an airtight container for further use. No other chemical or physical treatments were used prior to adsorption experiments.

2.3. Effect of PS adsorbent dose on the MB adsorption

To study the effect of PS dose on the MB adsorption, different amounts of PS (0.05, 0.10, 0.15, 0.20, 0.40, 0.60, 0.80 and 1.00 g) were added into a number of 250-mL stoppered glass (Erlenmeyers flasks) containing a definite volume (200 mL in each flask) of fixed initial concentration (50 mg/L) of dye solution without changing the solution pH at 30 °C. The flasks were placed in a thermostated water bath shaker (Protech, Malaysia) and agitation was provided at 130 rpm for 180 min and the dye concentrations were measured at equilibrium.

2.4. Effect of solution pH

In this study, the effect of solution pH on adsorption of MB on PS was studied by mixing 0.50 g of PS with 200 mL of dye solution of 50 mg/L initial concentration at different pH values (3.0–10.0) and 30 °C. The pH was adjusted with 0.1N NaOH and 0.1N HCl solutions and measured by using a pH meter (Ecoscan, EUTECH Instruments, Singapore). Agitation was made for 180 min at a constant agitation speed of 130 rpm. The dye concentrations were measured by a double beam UV–vis spectrophotometer (Shimadzu, Model UV 1601, Japan) at 668 nm wavelength. Prior to the measurement, a calibration curve was obtained by using the standard MB solution with the known concentrations.

2.5. Equilibrium studies

Batch adsorption experiments were carried out by adding a fixed amount of sorbent (0.50 g) into a number of 250-mL stoppered glass (Erlenmeyers flasks) containing a definite volume (200 mL in each case) of different initial concentrations (50–360 mg/L) of dye solution without changing pH and temperature 30 °C. The flasks were placed in a thermostatic water bath shaker and agitation was provided at 130 rpm for 180 min to ensure equilibrium was reached. The amount of adsorption at equilibrium, q_e (mg/g), was calculated by

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

where C_0 and C_e (mg/L) are the liquid-phase concentrations of dye at initial and equilibrium, respectively. V (L) is the volume of the solution and W (g) is the mass of dry sorbent used.

The dye removal percentage can be calculated as follows:

$$\text{Removal percentage} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

2.6. Batch kinetic studies

The procedures of kinetic experiments were basically identical to those of equilibrium tests. The aqueous samples were taken at preset time intervals, and the concentrations of dye were similarly measured. All the kinetic experiments were carried out at 30 °C. The amount of sorption at time t , q_t (mg/g), was calculated by

$$q_t = \frac{(C_0 - C_t)V}{W} \quad (3)$$

where C_t (mg/L) is the liquid-phase concentrations of dye at any time.

3. Theory of adsorption isotherm and kinetics

3.1. Isotherm models

Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. The Langmuir [19], the Freundlich [20] and the Temkin [21] were employed in the present study. The linearized forms of the three isotherms are

$$\frac{1}{q_e} = \frac{1}{K_a q_m C_e} + \frac{1}{q_m} \quad (4)$$

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (5)$$

$$q_e = B \ln A + B \ln C_e \quad (6)$$

where $B = RT/b$. The Langmuir constants q_m (mg/g) and K_a (L/mg) are Langmuir constants related to adsorption capacity and energy of adsorption, respectively. The constants q_m and K_a can be calculated from the plot between $1/q_e$ versus $1/C_e$ (Eq. (4)). C_e (mg/L) and q_e (mg/g) are the equilibrium concentration, and the amount of dye adsorbed at equilibrium, respectively. Similarly the Freundlich isotherm constants K_F and $1/n$ can be calculated from the plot of $\ln(q_e)$ versus $\ln(C_e)$ (Eq. (5)). K_F and n are the Freundlich constants, which are indicators of adsorption capacity and adsorption intensity, respectively [22]. The Temkin isotherm [21] has generally been applied in the form given by Eq. (6). Therefore, by plotting q_e versus $\ln C_e$ (Eq. (6)), enables the determination of the constants A and B . B is the Temkin constant related to heat of sorption (J/mol), A is the Temkin isotherm constant (L/g), R the gas constant (8.314 J/mol K), b is Temkin isotherm constant and T the absolute temperature (K).

3.2. Kinetics models

The most common models used to fit the kinetic sorption experiments are Lagergren's pseudo-first-order model (Eq. (7)) [23] and pseudo-second-order model (Eq. (8)) [24] were used:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (7)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (8)$$

where q_e (mg/g) and q_t (mg/g) are the amount of adsorbate adsorbed at equilibrium and at time t , respectively. k_1 (min^{-1}) and k_2 ($\text{g mg}^{-1} \text{min}$) are the pseudo-first-order and pseudo-second-order adsorption rate constants, respectively.

3.3. Intraparticle diffusion model

In order to investigate the mechanism of the MB adsorption onto PS, intraparticle diffusion-based mechanism was studied. The most commonly used technique for identifying the mechanism involved in the adsorption process is by fitting an intraparticle diffusion plot. It is an empirically found functional relationship, common to the most adsorption processes, where uptake varies almost proportionally with $t^{1/2}$ rather than with the contact time t . According to the theory proposed by Weber and Morris [25]:

$$q_t = k_{id} t^{1/2} + C_i \quad (9)$$

where k_{id} ($\text{mg g}^{-1} \text{min}^{1/2}$), the rate parameter of stage i , is obtained from the slope of the straight line of q_t versus $t^{1/2}$. C_i is the intercept.

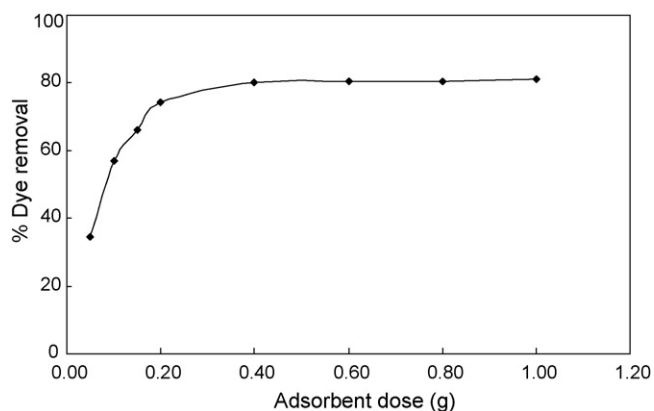


Fig. 1. Effect of adsorbent dosage on the adsorption of MB on PS ($T = 30^\circ\text{C}$ and $C_0 = 50\text{ mg/L}$).

4. Results and discussion

4.1. Effect of adsorbent dose on dye adsorption

Fig. 1 shows the effect of adsorbent dose (W (g)) on the removal of MB at $C_0 = 50\text{ mg/L}$ and 30°C . It can be seen that the MB removal increases with increase W up to 0.40 g, thereafter remained fairly constant despite increase in the amount of the PS to 1.0 g. At equilibrium time, the % removal increased from 34.67 to 80% for an increase in PS dose from 0.05 to 0.40 g. The increase in % removal was due to the increase of the available sorption surface and availability of more adsorption sites. A similar behavior was reported for the adsorption of methylene blue on peanut hull [26] and invasive marine seaweed (*Caulerpa racemosa* var. *cylindracea*) [27].

4.2. Effect of solution pH on dye adsorption

In this work, the effect of initial pH on equilibrium uptake of PS was studied at 50 mg/L initial MB concentration and 30°C . Fig. 2 shows that the sorption of MB was minimum at solution pH 3 and increased with pH up to 4.0 and then remained nearly constant over the initial pH ranges of 4–10. The observed low adsorption rate of MB on the PS at pH <4 may be because the surface charge become positively charged, thus making (H^+) ions compete effectively with dye cations causing a decrease in the amount of dye adsorbed. Similar results were reported for the adsorption of MB on wheat shells [28], fallen phoenix tree's leaves [29] and yellow passion fruit peel [9].

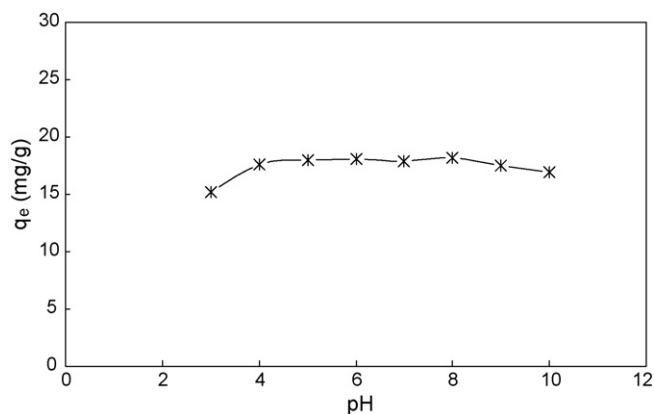


Fig. 2. Effect of pH on equilibrium uptake of MB ($W = 0.50\text{ g}$; $V = 0.20\text{ L}$; $C_0 = 50\text{ mg/L}$; $T = 30^\circ\text{C}$).

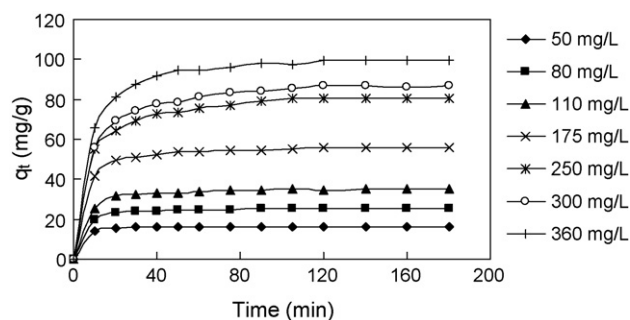


Fig. 3. Effect of initial concentration and contact time on MB adsorption ($W = 0.50\text{ g}$; $V = 0.20\text{ L}$; $T = 30^\circ\text{C}$).

4.3. Effect of contact time and initial concentration

Fig. 3 shows the result for effect of initial concentration (50–360 g/L) on adsorption of MB on PS at 30°C . It was observed that dye uptake was rapid for the first 20 min and thereafter it proceeded at a slower rate and finally reached saturation. The initial rapid phase may be due to an increase in the number of vacant sites available at the initial stage. Fig. 3 indicates that an increase in initial MB concentration leads to increase in the adsorption of MB on PS. The equilibrium adsorption increased from 16.42 to 99.30 mg/g, with increase in the initial MB concentration from 50 to 360 mg/L. As the initial MB concentration increased from 50 to 360 mg/L the equilibrium removal of MB decreased from 77.10 to 68.63%. Such trend was reported on the adsorption of MB peanut hull [26].

The adsorption of MB at low initial concentration (50–80 mg/L) reached equilibrium in less than 30 min, while at high initial MB concentration (110–360 mg/L), the time necessary to reach equilibrium was 120 min. However, the experimental data were measured at 180 min to be sure that full equilibrium was attained. Data on the adsorption kinetics of MB by various adsorbents have shown different ranges of adsorption rates. For example, the equilibrium times reported for the adsorption of MB were 150 and 90 min for the adsorption of MB on fallen phoenix tree's leaves [29] and marine seaweed (*C. racemosa* var. *cylindracea*) [27], respectively.

4.4. Isotherm analysis

Equilibrium data, commonly known as adsorption isotherms, are basic requirements for the design of adsorption systems [30]. In this work, the equilibrium data for MB on PS were modeled with the Langmuir, Freundlich and Temkin models. The linear plot of specific adsorption ($1/q_e$) against the equilibrium concentration ($1/C_e$) (Fig. 4) shows that the adsorption obeys the Langmuir model. The Langmuir constants q_m and K_a were determined from the slope and intercept of the plot and are presented in Table 1. The value of

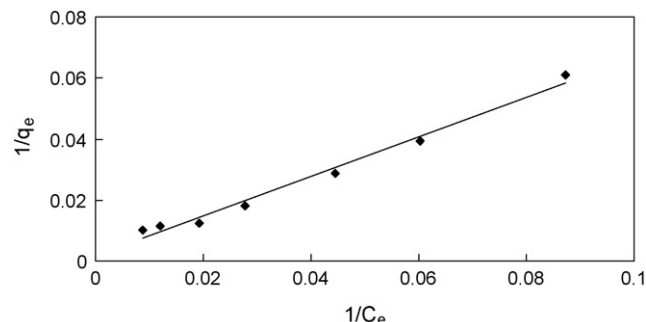


Fig. 4. Langmuir isotherm of MB on PS.

Table 1
Isotherm parameters for adsorption of MB by PS at 30 °C

Isotherm	Parameters	Values
Langmuir	q_m (mg g ⁻¹)	555.557
	K_a (L mg ⁻¹)	0.0028
	R^2	0.9863
Freundlich	K_F	2.806
	n	1.2633
	R^2	0.9507
Temkin	A (L g ⁻¹)	8.0418
	B	38.119
	R^2	0.9841

the coefficient of correlation ($R^2 = 0.9863$) obtained from Langmuir expression indicates that Langmuir expression provided a better fit to the experimental data of MB on PS.

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor R_L that is given by the following equation [31]:

$$R_L = \frac{1}{(1 + K_a C_0)} \quad (10)$$

where C_0 (mg/L) is the highest initial concentration of adsorbate, and K_a (L/mg) is Langmuir constant. The value of R_L indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$). The R_L value for the adsorption of MB onto PS was 0.498, indicating that the adsorption was a favorable process.

The equilibrium data were further analyzed using the linearized form of Freundlich isotherm, by plotting $\ln q_e$ versus $\ln C_e$ (Fig. 5). The calculated Freundlich isotherm constants and the corresponding coefficient of correlation are shown in Table 1. The coefficient of correlation was high ($R^2 = 0.9507$) showing a good linearity.

The result shows that the value of n is greater than unity ($n = 1.2633$) indicating that the dye is favorably adsorbed on PS. This is in great agreement with the findings regarding to R_L value. The magnitude of Freundlich constant indicates easy uptake of MB from aqueous solution.

The adsorption data for MB on PS were analyzed by a regression analysis to fit the Temkin isotherm model (Fig. 6). The parameters of Temkin model as well as the correlation coefficient are listed

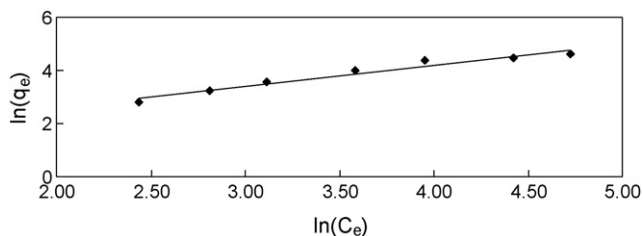


Fig. 5. Freundlich adsorption isotherm of MB on PS.

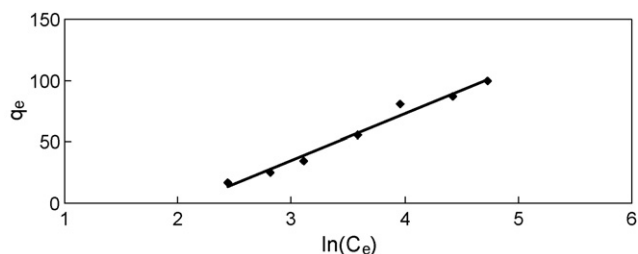


Fig. 6. Temkin adsorption isotherm of MB on PS.

Table 2
Comparison of adsorption capacities of various adsorbents for methylene blue

Adsorbent	Maximum adsorption capacity (mg g ⁻¹)	Reference
Papaya seeds	555.557	This work
Guava (<i>Psidium guajava</i>) leaf powder	185.2	[12]
Coffee husks	90.09	[14]
Pomelo (<i>Citrus grandis</i>) peel	344.83	[16]
<i>Caulerpa racemosa</i> var. <i>cylindracea</i>	3.423	[27]
Wheat shells	16.56	[28]
Yellow passion fruit waste	44.70	[9]
Dehydrated wheat bran carbon	185.2	[30]
<i>Luffa cylindrica</i> fibers	47	[32]
Dehydrated peanut hull	123.5	[33]
Palygorskite	50.8	[34]
<i>Euphorbia rigida</i> -based activated carbon	109.98	[35]
Durian shell-based activated carbon	289.26	[36]
Coir pith carbon	5.87	[37]
Rattan sawdust-based activated carbon	294.14	[38]
Bamboo-based activated carbon	454.2	[39]
Oil palm fibre-activated carbon	277.78	[40]
Oil palm shell-based activated carbon	243.90	[41]
<i>Hevea brasiliensis</i> seed coat-activated carbon	227.27	[42]

in Table 1. The coefficient of correlation was high ($R^2 = 0.9841$) showing a good linearity. From Table 1, it can be concluded that the Langmuir isotherm model was more suitable for the experimental data than other isotherms because of the high value of correlation coefficient ($R^2 = 0.9863$). This indicates that the adsorption of MB on PS takes place as monolayer adsorption on a surface that is homogenous in adsorption affinity. A similar result was reported for the adsorption of MB on *Luffa cylindrica* fibers [32].

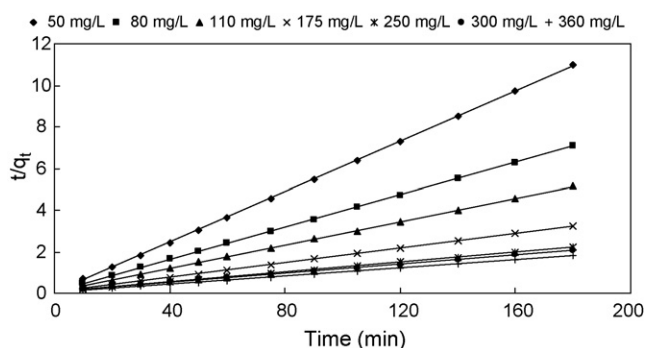
Table 2 lists a comparison of adsorption capacity of the PS with those obtained in the literature for the adsorption of MB. It can be seen that the PS is more effective for this purpose even when compared with activated carbons. The adsorption capacity of PS for MB was 555.557 mg/g which is higher than the adsorption capacities of various low-cost adsorbents and activated carbons for MB reported in the literature such as guava (*P. guajava*) leaf powder [12], coffee husks [14], pomelo (*C. grandis*) peel [16], *C. racemosa* var. *cylindracea* [27], wheat shells [28], Yellow passion fruit waste [9], dehydrated wheat bran carbon [30], *L. cylindrica* fibers [32], dehydrated peanut hull [33], palygorskite [34], activated carbon prepared from *Euphorbia rigida* [35], activated carbon prepared from durian shell [36], coir pith carbon [37], activated carbon prepared from rattan sawdust [38], bamboo-based activated carbon [39], oil palm fibre-activated carbon [40], activated carbon prepared from oil palm shell [41] and *Hevea brasiliensis* seed coat [42]. It can be concluded that PS was extremely effective for removal of MB from aqueous solution.

4.5. Adsorption kinetics

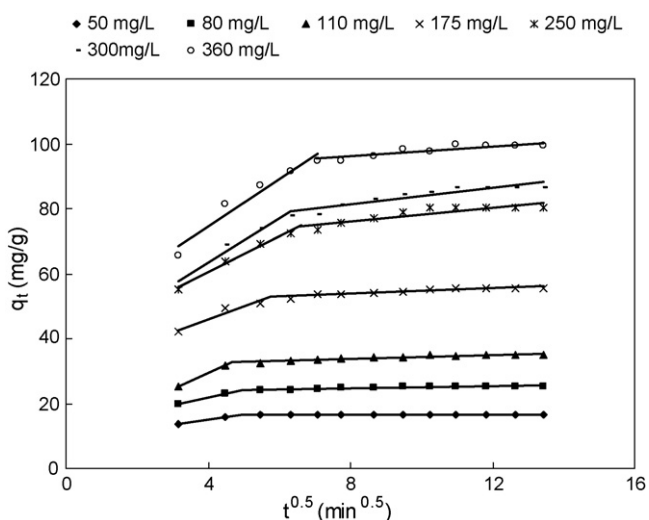
The kinetics of MB adsorption on PS was studied with respect to different initial concentrations. For evaluating the adsorption kinetics of MB, the pseudo-first-order and pseudo-second-order kinetic models were used to fit the experimental data. Using Eq. (7), $\log(q_e - q_t)$ versus t was plotted at different MB concentrations (figure not shown). The pseudo-first-order model data do not fall on straight lines for most initial concentrations indicating that this model is less appropriate. The Lagergren first-order rate constant (k_1) and $q_{e,cal}$ determined from the model are presented in Table 3 along with the corresponding correlation coefficients. It was found that the correlation coefficients for the pseudo-first-order model are low and a wide range of variations are recorded for the q_e , obtained from the experimental and calculated. This indicates that the adsorption of MB on PS does not follow pseudo-first-order kinetics. Therefore, the experimental kinetic data were

Table 3Comparison of the pseudo-first-order and pseudo-second-order adsorption rate constants, and calculated and experimental q_e values for different initial MB concentrations

Initial concentration (mg L^{-1})	$q_{e,\text{exp}}$ (mg g^{-1})	Pseudo-first-order kinetic			Pseudo-second-order kinetic		
		k_1 (min^{-1})	$q_{e,\text{cal}}$ (mg g^{-1})	R^2	k_2 ($\text{g mg}^{-1} \text{min}$)	$q_{e,\text{cal}}$ (mg g^{-1})	R^2
50	16.420	0.1806	14.588	0.8912	0.0920	16.529	0.9999
80	25.354	0.0396	5.853	0.9527	0.01628	25.773	0.9999
110	35.000	0.0309	7.663	0.8782	0.0088	35.714	0.9999
175	55.600	0.0408	18.689	0.8921	0.00538	56.818	1.0000
250	80.400	0.0274	14.710	0.8287	0.00206	83.333	0.9998
300	86.600	0.0304	34.483	0.9871	0.00175	90.090	0.9998
360	99.298	0.0283	29.902	0.8486	0.00198	103.093	0.9998

**Fig. 7.** Pseudo-second-order kinetic for adsorption of MB on PS.

further analyzed using the pseudo-second-order model. By plotting t/q_t against t for different initial MB concentrations (Fig. 7), a straight line was obtained in all cases and using Eq. (8) the second-order rate constant (k_2) and q_e were determined from the plots. The $q_{e,\text{exp}}$ and the $q_{e,\text{cal}}$ values along with correlation coefficients for the pseudo-second-order models are shown in Table 3. The values of correlation coefficient were very high ($R^2 > 0.999$) and the theoretical $q_{e,\text{cal}}$ values were closer to the experimental $q_{e,\text{exp}}$ values at different initial MB concentrations (Table 3). From Table 3, it can be concluded that the pseudo-second-order kinetic model provided a good correlation for the adsorption of MB on PS at different initial MB concentrations compared to the pseudo-first-order model. A number of authors have reported pseudo-second-order kinetics for adsorption of methylene blue on wheat shells [28], palygorskite [34] and dehydrated wheat bran carbon [30].

**Fig. 8.** Intraparticle diffusion plot for adsorption of MB on PS for different initial MB concentrations.

The pseudo-first-order and pseudo-second-order kinetic models could not identify the diffusion mechanism. Thus the kinetic results were then analyzed by using the intraparticle diffusion model. Weber and Moris plot [25] (q_t versus $t^{0.5}$) was used to investigate intraparticle diffusion mechanism (Fig. 8). If the value of C_i is zero, then the rate of adsorption is controlled by intraparticle diffusion for the entire adsorption period. However, the plot of q_t against $t^{0.5}$ usually shows more than one linear portion. As seen from Fig. 8, the plots were not linear over the whole time range, implying that more than one process affected the adsorption. A similar behavior was reported for methylene blue adsorption onto palm kernel fibre [43].

5. Conclusions

The adsorption of MB from aqueous solution using papaya seeds as low-cost adsorbent was investigated under different experimental conditions in batch process. The Langmuir adsorption isotherm was found to have the best fit to the experimental data with maximum adsorption capacity of 555.557 mg/g. The adsorption kinetics can be predicted by pseudo-second-order kinetic. The results of the present investigation indicated that PS, a low-cost adsorbent could be employed as an alternative to commercial-activated carbon for the removal of MB from aqueous solutions.

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