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Adsorption of basic dye (methylene blue) onto activated carbon prepared from rattan sawdust

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Abstract

Activated carbon prepared from non-wood forest product waste (rattan sawdust) has been utilized as the adsorbent for the removal of methylene blue dye from an aqueous solution. The experimental data were analyzed by the Langmuir and Freundlich models of adsorption. Equilibrium data fitted well with the Langmuir model with maximum monolayer adsorption capacity of 294.14 mg/g. The dimensionless factor, R_L revealed the favorable nature of the isotherm of the dye–activated carbon system. The rates of adsorption were found to conform to the pseudo-second-order kinetics with good correlation. The kinetic parameters of this best-fit model were calculated and the results are discussed.

Keywords: Adsorption; Activated carbon; Isotherm; Methylene blue; Kinetics; Rattan sawdust

1. Introduction

Textile industries in Malaysia use dyes or pigments to color their final products. For example, methylene blue, which is the most common among all other dyes of its category, is generally used for dyeing cotton and silk. Since dyes have a synthetic origin and complex aromatic molecular structures, are inert and difficult to biodegrade when discharged into waste streams. This aspect has always been overlooked in their discharge [1].

From an environmental point of view, the removal of synthetic dyes is of great concern, since some dyes and their degradation products may be carcinogens and toxic and, consequently, their treatment cannot depend on biodegradation alone [2,3]. Though methylene blue is not strongly hazardous, it can cause some harmful effects. Acute exposure to methylene blue can cause increased heart rate, vomiting, shock, Heinz body formation, cyanosis, jaundice, quadriplegia, and tissue necrosis in humans [4]. Therefore, the removal of such dye from process effluent becomes environmentally important.

Among several chemical and physical methods, the adsorption onto activated carbon has been found to be superior to other techniques for removal of dyes from aqueous solution in terms of methodology, its capability for efficiently adsorbing a broad range of different types of adsorbates and simplicity of design of adsorber. Commercially available activated carbons are usually derived from natural materials such as wood or coal, and therefore, are still considered expensive [5]. This has led to the search for cheaper substitutes. Hence, low-cost activated carbons based on agricultural solid wastes are investigated for a long time. Agricultural byproducts and waste materials used for the production of activated carbons include plum kernels [6], cassava peel [7], bagasse [8], jute fiber [9], palm-tree cobs [10], rice husks [11], olive stones [12], date pits [13], fruit stones and nutshells [14].

Rattan (family *Palmae/Arecaceae*) is a spiny climbing plant belonging to the palm family. It is considered to be the most important non-wood forest product in Peninsular Malaysia. There are about 600 species in the world, of which 106 species are found in Peninsular Malaysia. Only 21 of these species, however, are utilized and marketed [15]. The stems of the climbing palms or rattans of Southeast Asia are an important natural resource used primarily for the production of cane

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Nomenclature

b	adsorption energy constant of Langmuir adsorption isotherm (L/mg)
C_e	equilibrium liquid-phase concentration (mg/L)
C_0	initial liquid-phase concentration (mg/L)
K_F	Freundlich isotherm constant related to adsorption capacity ((mg/g) (L/mg)$^{1/n}$)
k_1	rate constant of first-order adsorption (1/min)
k_2	rate constant of second-order adsorption (g/mg min)
n	Freundlich isotherm constant related to adsorption intensity
Q	the maximum surface coverage (formation of monolayer) of sorbent (mg/g)
q_e	equilibrium solid-phase adsorbate concentration (mg/g)
q_t	amount of adsorption at time t (mg/g)
R^2	correlation coefficient
R_L	dimensionless separation factor
V	volume of solution (L)
W	mass of adsorbent (g)

furniture [16]. It also plays an important role in the manufacture of household commodities in many rural areas [17]. There are 653 rattan mills throughout the country manufacturing rattan furniture and rattan products such as walking sticks, rattan balls, baskets, toys and mats [18]. The use of rattan sawdust (RSD) as feedstock for activated carbon production would add value to the rattan sawdust, turn the sawdust into value-added commodities. Furthermore, conversion of rattan sawdust to a value-added product such as activated carbon will help to solve part of the problem of textile wastewater treatment in Malaysia. The advantage of using non-wood forest products as raw materials for manufacturing activated carbon is that these raw materials are renewable and potentially less expensive to manufacture.

The purpose of this work was to prepare activated carbon from rattan sawdust and to find out the possibility of using this activated carbon as low-cost adsorbent for the removal of methylene blue dye from aqueous solution. The equilibrium and kinetic data of the adsorption were then studied to understand the adsorption.

2. Materials and methods

2.1. Adsorbate: methylene blue

Methylene blue (MB) supplied by Sigma–Aldrich (M) Sdn Bhd, Malaysia was used as an adsorbate and was not purified prior to use. Double distilled water was employed for preparing all the solutions and regents. MB has a molecular weight of 373.9 g/mol, which corresponds to methylene blue hydrochloride with three groups of water. The chemical structure of the dye is shown in Appendix A.

2.2. Preparation and characterization of activated carbon

The rattan sawdust was collected from a local furniture factory. It was washed with hot distilled water to remove dust like impurities, dried and the material was finally sieved to discrete sizes. The raw material was then carbonized at 700 °C under nitrogen atmosphere for 1 h. A certain amount of produced char then was soaked with potassium hydroxide (KOH) at impregnation ratio of 1:1. The mixture was dehydrated in an oven overnight at 105 ± 1 °C, then pyrolysed in a stainless steel vertical tubular reactor placed in a tube furnace under high-purity nitrogen (99.995%) flow of 150 cm^3/min to a final temperature of 850 °C and 2 h soaking. Once the final temperature was reached, the nitrogen gas flow was switched to carbon dioxide and activation was continued for 2 h. The activated product (RSD-AC) was then cooled to room temperature and washed with deionized water to remove remaining chemical. Subsequently the sample was transferred to a beaker containing 250-mL solution of hydrochloric acid (about 0.1 mol/L), stirred for 1 h, and then washed with hot deionized water.

Textual characterization of the activated carbon was carried out by N_2 adsorption at 77 K using Autosorb I, supplied by Quantachrome Corporation, USA. The Brunauer–Emmett–Teller (BET) (N_2, 77 K) is the most usual standard procedure used when characterizing an activated carbon [19]. It was found that the BET surface area, average pore diameter and pore volume of the activated carbon were 1083 m^2/g, 2.77 nm and 0.644 cm^3/g, respectively.

2.3. Analysis of the samples

The concentration of methylene blue in the supernatant solution before after and adsorption was determined using a double beam UV–vis spectrophotometer (Shimadzu, Japan) at 668 nm. It was found that the supernatant from the activated carbon did not exhibit any absorbance at this wavelength and also that the calibration curve was very much reproducible and linear over the concentration range used in this work.

2.4. Batch equilibrium studies

Adsorption isotherms were performed in a set of 43 Erlenmeyer flasks (250 mL), where solutions of dye (100 mL) with different initial concentrations (100–500 mg/L) were placed in these flasks. Equal masses of 0.1 g of activated carbon of particle size (150 μm) were added to dye solutions and each sample was kept in an isothermal shaker (30 ± 1 °C) for 24 h to reach equilibrium of the solid–solution mixture. Similar procedure was followed for another set of Erlenmeyer flask containing the same dye concentration without activated carbon to be used as a blank. The flasks were then removed

from the shaker and the final concentration of dye in the solution was analyzed. Each experiment was duplicated under identical conditions. 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 is the volume of the solution (L) and W is the mass of dry adsorbent used (g).

2.5. Batch kinetic studies

The procedures of kinetic experiments were basically identical to those of equilibrium tests. The aqueous samples were taken at different time intervals and the concentrations of dye were similarly measured. The amount of adsorption at time t, q_t (mg/g), was calculated by:

$$q_t = \frac{(C_0 - C_t)V}{W} \quad (2)$$

where C_0 and C_t (mg/L) are the liquid-phase concentrations of dye at initial and at any time t, respectively. V is the volume of the solution (L) and W is the mass of dry adsorbent used (g).

3. Results and discussion

3.1. Effect of initial dye concentration on adsorption

To determine proper methylene blue adsorption and equilibrium time, initial concentrations of methylene blue solutions were changed and time intervals were assessed until no adsorption of adsorbate onto RSD-AC took place. The adsorption data for the uptake of methylene blue versus contact time at different concentrations are presented in Fig. 1. The results indicate that the actual amount of MB adsorbed per unit mass of RSD-AC increased with increase in MB concentration. This is due to increase in the driving force of the concentration gradient, as an increase in the initial dye concentration. The unit adsorption for MB increased from 96 to 294 mg/g as the MB concentration increased from 100 to 500 mg/L. It is also evident from Fig. 1 that the contact time needed for MB solutions with initial concentrations of 100–200 mg/L to reach equilibrium was less than 4 h. However, for MB solutions with higher initial concentrations, longer equilibrium times were required.

These results also indicate that the sorption process can be considered very fast because a significant amount of MB was adsorbed onto the sorbent within the first 5 h of adsorption. During adsorption of MB, initially the dye molecules reached the boundary layer, then they had to diffuse into the adsorbent surface, and finally, they had to diffuse into the porous structure of adsorbent. Hence, this phenomenon will take a relatively longer contact time [9].

3.2. Adsorption isotherms

The adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. The analysis of the isotherm data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purpose [20]. The amount of MB dye adsorbed (q_e) has been plotted against the equilibrium concentration (C_e) as shown in Fig. 2. The equilibrium adsorption density, q_e increased with the increase in dye concentration.

Several models have been published in the literature to describe the experimental data of adsorption isotherms. The Langmuir and Freundlich are the most frequently employed models. In this work, both models were used to describe the relationship between the amount of dye adsorbed and its equilibrium concentration.

3.2.1. Langmuir isotherm

Langmuir's isotherm model suggests that uptake occurs on homogeneous surface by monolayer sorption without interaction between sorbed molecules. The model assumes uniform

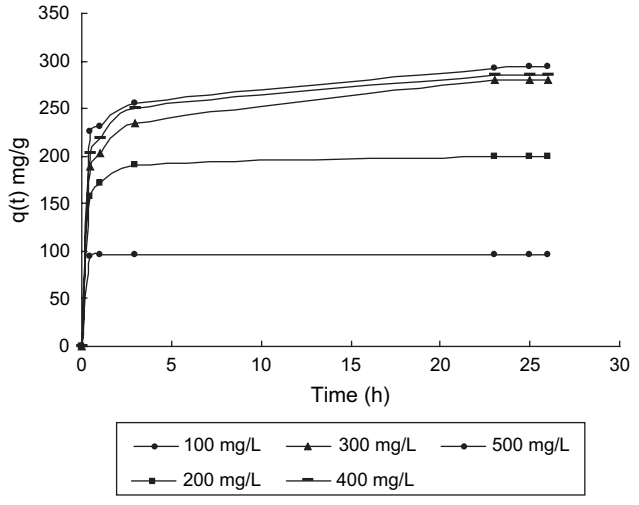

Fig. 1. Effect of initial dye concentration on the adsorption of MB onto the prepared activated carbon.

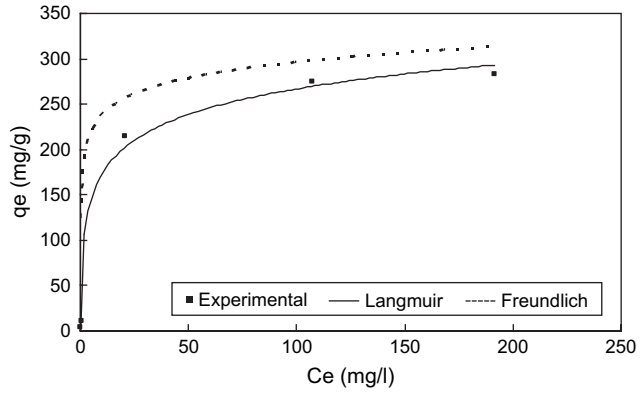

Fig. 2. Equilibrium adsorption isotherm of methylene blue onto activated carbon.

energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface. The linear form of Langmuir isotherm equation is represented by the following equation [21]:

$$\frac{C_e}{q_e} = \frac{1}{bQ} + \frac{C_e}{Q} \tag{3}$$

where q_e is the amount adsorbed at equilibrium time (mg/g), C_e is the equilibrium concentration of the adsorbate ions (mg/L), Q and b are Langmuir constants related to maximum adsorption capacity (monolayer capacity) and energy of adsorption, respectively. When C_e/q_e is plotted against C_e, a straight line with slope $1/Q$ and intercept $1/bQ$ is obtained (Fig. 3a), which shows that the adsorption of MB follows Langmuir isotherm model.

Conformation of the experimental data with the Langmuir isotherm model indicates the homogeneous nature of rattan sawdust carbon surface, i.e., each dye molecule/RSD-AC adsorption has equal adsorption activation energy; the results also demonstrate the formation of monolayer coverage of dye molecule at the outer surface of prepared RSD-AC. Similar observation was reported by the adsorption of acid orange 10 dye onto activated carbon prepared from agricultural waste bagasse [8], the adsorption of direct dyes onto activated carbon prepared from sawdust [22] and the adsorption of congo red dye onto activated carbon from coir pith [23]. Values of Q and b were calculated from the intercept and slope of the linear plot and are presented in Table 1.

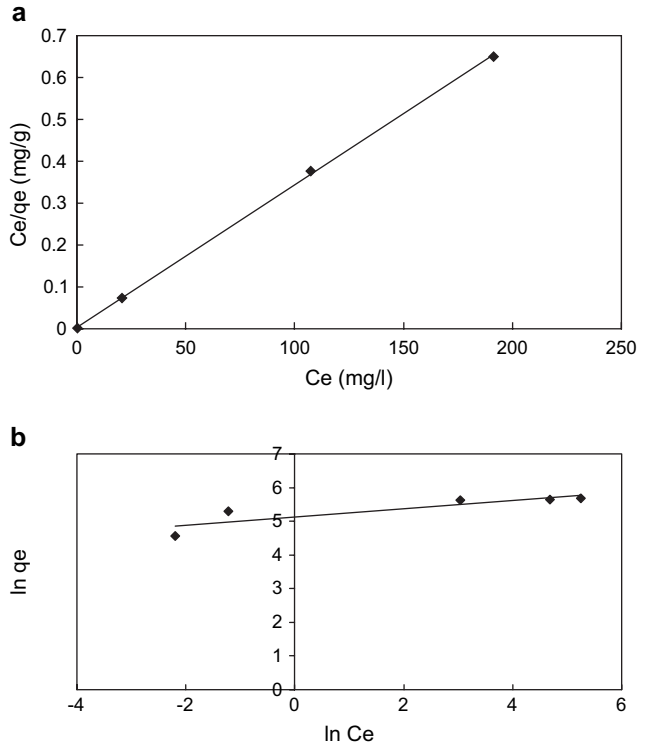

Fig. 3. (a) Linearized Langmuir isotherm for dye adsorption by activated carbon. (b) Linearized Freundlich isotherm for dye adsorption by activated carbon.

Table 1
Langmuir and Freundlich isotherm constants for MB dye on rattan-based activated carbon at 30 °C

Langmuir isotherm			Freundlich isotherm		
Q (mg/g)	b (L/mg)	R^2	n	K_F (mg/g) (L/mg)$^{1/n}$	R^2
294.12	0.131	0.999	8.22	169.9	0.76

The essential characteristics of the Langmuir equation can be expressed in terms of a dimensionless separation factor, R_L, defined as [24]:

$$R_L = \frac{1}{(1 + bC_0)} \tag{4}$$

where C_0 is the highest initial solute concentration, b is the Langmuir's adsorption constant (L/mg). The R_L value implies the adsorption to be unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). Value of R_L was found to be 0.015 and confirmed that the prepared activated carbon is favorable for adsorption of methylene blue dye under conditions used in this study.

3.2.2. Freundlich isotherm

The Freundlich isotherm [25] is the earliest known relationship describing the sorption equation. This fairly satisfactory empirical isotherm can be used for non-ideal sorption that involves heterogeneous sorption and is expressed by the following equation:

$$q_e = K_F C_e^{1/n} \tag{5}$$

The equation may be linearized by taking the logarithm of both sides:

$$\ln(q_e) = 1/n \ln(C_e) + \ln(K_F) \tag{6}$$

where q_e is the amount adsorbed (mg/g), C_e is the equilibrium concentration of the adsorbate (MB), K_F and n are Freundlich constants, n giving an indication of how favorable the adsorption process is and K_F (mg/g)(L/mg)$^{1/n}$ is the adsorption capacity of the adsorbent. K_F can be defined as the adsorption or distribution coefficient and represents the quantity of dye adsorbed onto activated carbon adsorbent for a unit equilibrium concentration. Fig. 3b shows a straight line with a slope of $1/n$ and an intercept of ln (K_F) when ln (q_e) is plotted against ln (C_e). The slope $1/n$ ranging between 0 and 1, is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero [26]. A value of $1/n$ below one indicates a normal Langmuir isotherm while $1/n$ above one is indicative of cooperative adsorption [27]. Freundlich constants (K_F and n) were calculated and recorded, which are listed in Table 1. The results suggest that MB is favorably adsorbed by RSD-AC. The Langmuir isotherm fits quite well with the experimental data (correlation coefficient $R^2 = 0.999$), whereas the low correlation coefficient ($R^2 = 0.76$) shows poor agreement of Freundlich isotherm with the experimental data. The experimental data of

MB dye/RSD-AC equilibrium isotherm as shown in Fig. 2 was compared using two isotherms namely, Langmuir and Freundlich.

Table 2 lists a comparison of maximum monolayer adsorption capacity of some dyes with various adsorbents. Table 2 shows that the RSD-AC studied in this work has very large adsorption capacity. This is due to its high surface area (1083 m^2/g).

3.3. Adsorption kinetics

Kinetic adsorption data were treated with pseudo-first-order kinetic model [29]:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{7}$$

where q_e and q_t refer to the amount of dye adsorbed (mg/g) at equilibrium and at any time, t (min), respectively, and k_1 is the equilibrium rate constant of pseudo-first-order sorption (1/min). Integration of Eq. (7) for the boundary conditions $t = 0$ to t and $q_t = 0$ to q_t, gives:

$$\log\frac{q_e}{(q_e - q_t)} = \frac{k_1}{2.303}t \tag{8}$$

which is the integrated rate law for a pseudo-first-order reaction. Eq. (8) can be rearranged to obtain a linear form:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \tag{9}$$

The slope and intercept of the plot of log $(q_e - q_t)$ versus t were used to determine the first-order rate constant, k_1 (Fig. 4). In many cases, the first-order equation of Lagergren does not fit well with the whole range of contact time and is generally applicable over the initial stage of the adsorption processes [30].

Kinetic data were further treated with the pseudo-second-order kinetic model [31]. The differential equation is the following:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{10}$$

where k_2 is the equilibrium rate constant of pseudo-second-order adsorption (g/mg min). Integrating Eq. (10) for the boundary condition $t = 0$ to t and $q_t = 0$ to q_t, gives the following:

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + k_2 t \tag{11}$$

which is the integrated rate law for a pseudo-second-order reaction. Eq. (11) can be rearranged to obtain a linear form:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{12}$$

The slope and intercept of the plot of t/q_t versus t were used to calculate the second-order rate constant, k_2 (Fig. 5). It is more likely to predict the behavior over the whole range of adsorption for the case of chemisorption mechanism as the rate-controlling step [30,31].

Table 3 lists the results of the rate constant studies for different initial dye concentrations by the pseudo-first-order and second-order models. The correlation coefficient, R^2 for the pseudo-second-order adsorption model has high value (>99%), and its calculated equilibrium adsorption capacity, $q_{e,cal}$ is consistent with experimental data. These facts suggest that the pseudo-second-order adsorption mechanism is predominant, and that the overall rate of the dye adsorption process appears to be controlled by the chemisorption process [30,31]. Similar phenomena had been observed in the adsorption of direct dyes onto activated carbon prepared from sawdust

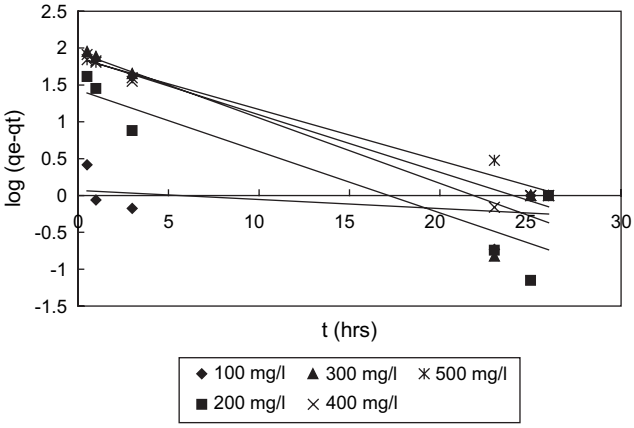

Fig. 4. Pseudo-first-order kinetics for the adsorption of MB dye onto activated carbon.

Table 2
Comparison of the maximum monolayer adsorption of some dyes onto various adsorbents

Dyes	Adsorbent	Maximum monolayer adsorption capacity, (mg/g)	Reference
Methylene blue	Rattan sawdust-activated carbon	294.12	This work
Acid yellow 36	Sawdust-activated carbon	183.8	[28]
Acid yellow 36	Rice husk-activated carbon	86.9	[28]
Congo red	Coir pith-activated carbon	6.72	[23]
Methylene blue	Jute fiber carbon	225.64	[9]
Methylene blue	Almond shell-activated carbon	1.33	[14]
Methylene blue	Walnut shell-activated carbon	3.53	[14]
Methylene blue	Hazelnut shell-activated carbon	8.82	[14]
Methylene blue	Apricot stones-activated carbon	4.11	[14]

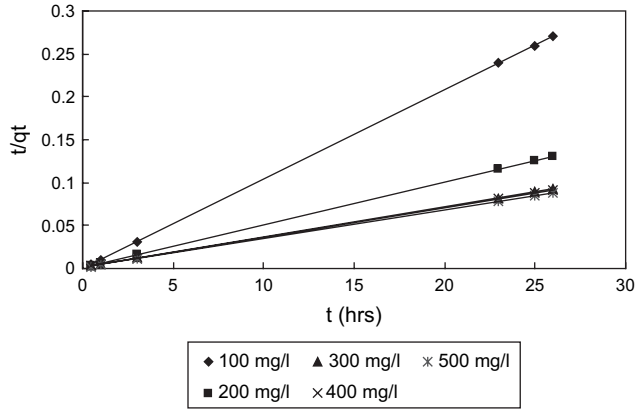

Fig. 5. Pseudo-second-order kinetics for the adsorption of MB dye onto activated carbon.

[22] and adsorption of congo red dye onto activated carbon from coir pith [23]. For the pseudo-second-order model, the data of which are presented in Table 3, the rate constant decreased with an increase of the initial dye concentration.

4. Conclusions

Activated carbon prepared from rattan sawdust was successfully employed as an adsorbent for the quantitative removal of methylene blue from aqueous solution. The equilibrium adsorption is practically achieved in less than 24 h. The equilibrium data fitted well in the Langmuir model of adsorption, showing monolayer coverage of dye molecules at the outer surface of sawdust carbon. Value of R_L was found to be 0.015 and confirmed that the prepared activated carbon is favorable for adsorption of MB blue dye. The kinetics of MB adsorption onto RSD-AC followed the pseudo-second-order model. These results indicated that the rattan-based activated carbon could be employed as a low-cost alternative to commercial activated carbon in the removal of methylene blue dye from wastewater.

Acknowledgements

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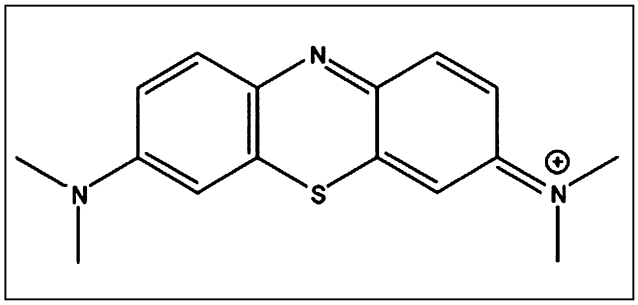
Table 3
Comparison of the pseudo-first- and second-order adsorption rate constants, and calculated and experimental q_e values for different initial dye concentrations

C_0 (mg/L)	First-order kinetic model				Second-order kinetic model		
	$q_{e,exp}$ (mg/g)	$q_{e,cal}$ (mg/g)	k_1 (1/min)	R^2	$q_{e,cal}$ (mg/g)	k_2 (g/mg min)	R^2
100	96.29	1.17	0.028	0.17	96.15	0.5410	1
200	199.24	20.89	0.192	0.85	200.00	0.0357	1
300	279.79	87.09	0.204	0.89	285.71	0.0182	0.99
400	285.20	74.13	0.179	0.97	285.71	0.0105	1
500	294.86	72.61	0.159	0.98	294.11	0.0102	0.99

Appendix A. Chemical structure of methylene blue dye



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