Adsorption of methylene blue onto bamboo-based activated carbon: Kinetics and equilibrium studies

B.H. Hameed [*], A.T.M. Din, A.L. Ahmad

School of Chemical Engineering, University Science Malaysia, Engineering Campus, 14300 Nibong Tebal, Penang, Malaysia

Abstract

Bamboo, an abundant and inexpensive natural resource in Malaysia was used to prepare activated carbon by physiochemical activation with potassium hydroxide (KOH) and carbon dioxide (CO_2) as the activating agents at 850 °C for 2 h. The adsorption equilibrium and kinetics of methylene blue dye on such carbon were then examined at 30 °C. Adsorption isotherm of the methylene blue (MB) on the activated carbon was determined and correlated with common isotherm equations. The equilibrium data for methylene blue adsorption well fitted to the Langmuir equation, with maximum monolayer adsorption capacity of 454.2 mg/g. Two simplified kinetic models including pseudo-first-order and pseudo-second-order equation were selected to follow the adsorption processes. The adsorption of methylene blue could be best described by the pseudo-second-order equation. The kinetic parameters of this best-fit model were calculated and discussed.
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Keywords: Activated carbon; Bamboo; Methylene blue; Isotherm; Kinetics

1. Introduction

Dyes are widely used by textile industries to color their products. One of the major problems concerning textile wastewaters is colored effluent. This wastewater contains a variety of organic compounds and toxic substances, which are harmful to fish and other aquatic organisms [1]. Methylene blue (MB) dye causes eye burns, which may be responsible for permanent injury to the eyes of human and animals. On inhalation, it can give rise to short periods of rapid or difficult breathing, while ingestion through the mouth produces a burning sensation and may cause nausea, vomiting, profuse sweating, mental confusion, painful micturition, and methemoglobinemia [2,3]. Therefore the treatment of effluent containing such dye is of interest due to its esthetic impacts on receiving waters.

Adsorption processes using activated carbons are widely used to remove pollutants from wastewaters. However, commercially available activated carbon is expensive. In the last years, special emphasis on the preparation of activated carbons from several agricultural by-products has been given due to the growing interest in low cost activated carbons from renewable, copious, especially for application concerning treatment of wastewater. Researchers have studied the production of activated carbon from palm-tree cobs [3], plum kernels [4], cassava peel [5], bagasse [6], jute fiber [7], rice husks [8], olive stones [9], date pits [10], fruit stones and nutshells [11]. The advantage of using agricultural by-products as raw materials for manufacturing activated carbon is that these raw materials are renewable and potentially less expensive to manufacture.

Plant biomass is a natural renewable resource that can be converted into useful materials and energy [12]. The idea of using bamboo to produce activated carbon rises in this context. Bamboo is a grass, the most diverse group of plants in the grass family. It belongs to the sub-family Bambusoidae of the family Poaceae (Graminae). Approximately 1500 commercial applications of bamboo have been identified—mostly in Asia [13]. It is an enduring, versatile, and highly renewable material, one that people and communities have known and utilized for thousands of years. Bamboo is an abundant natural resource in Malaysia because it takes only several months to grow up. It has been traditionally used to construct various living facilities and tools

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Nomenclature

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

Subscripts

exp	experimental
cal	calculated

[14]. Bamboo has been used as the structural material for steps at construction sites in China, India, Malaysia and other countries because it is a strong, tough and low-cost material. Conversion of bamboo to a value-added product such as activated carbon will help to solve part of the problem of wastewater treatment in Malaysia.

The focus of the research is to evaluate the adsorption potential of bamboo-based activated carbon for methylene dye due to the fact that the bamboo is a very abundant and inexpensive material. Methylene blue was chosen in this study because of its known strong adsorption onto solids and is often serves as a model compound for removing organic contaminants and colored bodies from aqueous solutions. MB which is the most commonly used material for dying cotton, wood, and silk has a molecular weight of $373.9\,g\,mol^{-1}$, which corresponds to methylene blue hydrochloride with three groups of water. The kinetic data and equilibrium data of adsorption studies were processed to understand the adsorption mechanism of the dye molecules onto the activated carbon.

2. Materials and methods

2.1. 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. Chemical structure of the dye is shown in Appendix A.

2.2. Preparation and characterization of activated carbon

Raw material (bamboo) used for preparation of activated carbon was procured locally, washed, dried, crushed to desired mesh size (1–2 mm). The raw material was then carbonized at 700 °C under nitrogen atmosphere for 1 h (first pyrolysis). A certain amount of produced char then was soaked with potassium hydroxide (KOH) at impregnation ratio of 1:1 (KOH pallets:char). The mixture was dehydrated in an oven overnight at 105 °C; then pyrolysed in a stainless steel vertical tubular reactor placed in a tube furnace under high purity nitrogen (99.995%) flow of $150\,cm^3\,min^{-1}$ (second pyrolysis) to a final temperature of 850 °C and activated for 2 h. Once the final temperature was reached, the gas flow was switched to carbon dioxide and activation was continued for 2 h. The activated product was then cooled to room temperature under nitrogen flow and washed with deionized water to remove remaining chemical. Subsequently the sample was transferred to a beaker containing a 250 ml solution of hydrochloric acid (about $0.1\,mol\,l^{-1}$), stirred for 1 h, and then washed with hot deionized water until the pH of the washing solution reached 6–7.

Textural characterization of the activated carbon (AC) was carried out by N_2 adsorption at 77 K using Autosorb I, supplied by Quantachrome Corporation, USA. The BET (N_2, 77 K) is the most usual standard procedure used when characterizing an activated carbon [15].

2.3. Analysis of methylene blue

The concentration of methylene blue in the supernatant solution after and before adsorption was determined using a double beam UV 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 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 (200 ml) with different initial concentrations ($100–500\,mg\,l^{-1}$) were placed in these flasks. Equal mass of 0.2 g of particle size (150 μm) activated carbon was added to dye solutions and kept in an isothermal shaker (30 ± 1 °C) for 48 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 pH was adjusted to 7 by adding either few drops of diluted hydrochloric acid or sodium hydroxide ($0.1\,mol\,l^{-1}$). The flasks were then removed from the shaker and the final concentration of dye in the solution was analyzed. The samples were filtered prior to analysis in order to minimize interference of the carbon fines with the analysis. Each experiment was duplicated under

identical conditions. The amount of adsorption at equilibrium, q_e (mg g^{-1}), was calculated by:

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

where C_0 and C_e (mg l^{-1}) 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 preset time intervals, and the concentrations of dye were similarly measured. The amount of adsorption at time t, q_t (mg g^{-1}), was calculated by:

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

where C_0 and C_t (mg l^{-1}) are the liquid-phase concentrations of dye at initial and 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. Textural characteristics of the prepared activated carbon

It was found that the BET surface area, total pore volume and average pore diameter of the activated carbon were 1896 m^2 g^{-1}, 1.109 cm^3 g^{-1} and 2.34 nm, respectively.

3.2. Effect of agitation time and concentration of dye on adsorption

Adsorption isotherms are usually determined under equilibrium conditions. A series of contact time experiments for MB dye have been carried out at different initial concentration (100–500 mg l^{-1}) and at temperature of 30 °C. Fig. 1 shows the contact time necessary for MB dye with initial concentrations of 100–300 mg l^{-1} to reach equilibrium is 6 h. However, for MB dye with higher initial concentrations (400–500 mg l^{-1}), longer equilibrium time of 24 h is needed. As can be seen from Fig. 1, the amount of the adsorbed dye onto activated carbon increases with time and, at some point in time, reaches a constant value beyond which no more is removed from solution. At this point, the amount of the dye desorbing from the adsorbent is in a state of dynamic equilibrium with the amount of the dye being adsorbed onto the activated carbon. The time required to attain this state of equilibrium is termed the equilibrium time, and the amount of dye adsorbed at the equilibrium time reflects the maximum adsorption capacity of the adsorbent under those operating conditions.

The adsorption capacity at equilibrium increases from 100 to 420 mg g^{-1} with an increase in the initial dye concentration from 100 to 500 mg l^{-1}. It is evident that the activated carbon
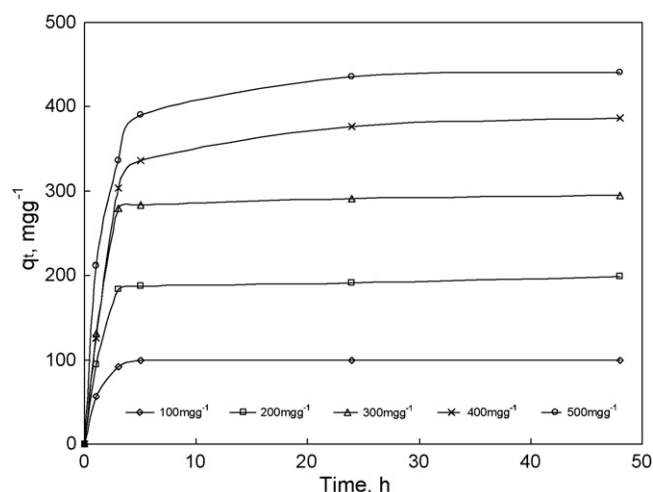

Fig. 1. The variation of adsorption capacity with adsorption time at various initial dye concentrations at 30 °C (pH 7, $W = 0.2$ g).

prepared from bamboo is efficient to adsorb MB dye from aqueous solution, the process attaining equilibrium gradually. This is due to the fact that activated carbon is composed of porous structure with large internal surface area (1896 m^2 g^{-1}). Three consecutive mass transport steps are associated with the adsorption of solute from solution by porous adsorbent [16]. First, the adsorbate migrates through the solution, i.e. film diffusion, followed by solute movement from particle surface into interior site by pore diffusion and finally the adsorbate is adsorbed into the active sites at the interior of the adsorbent particle. This phenomenon takes relatively long contact time. A similar phenomenon was observed for the adsorption of methylene blue from aqueous solution on jute fiber carbon and the equilibrium time was 250 min [7].

3.3. 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 [17].

Fig. 2 typically shows the adsorption isotherms of MB dye at 30 °C on the activated carbon. Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. Adsorption isotherm study is carried out on two well-known isotherms, Langmuir and Freundlich. Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface [18]. While, Freundlich isotherm model assumes heterogeneous surface energies, in which the energy term in Langmuir equation varies as a function of the surface coverage [18]. The applicability of the isotherm equation is compared by judging the correlation coefficients, R^2.
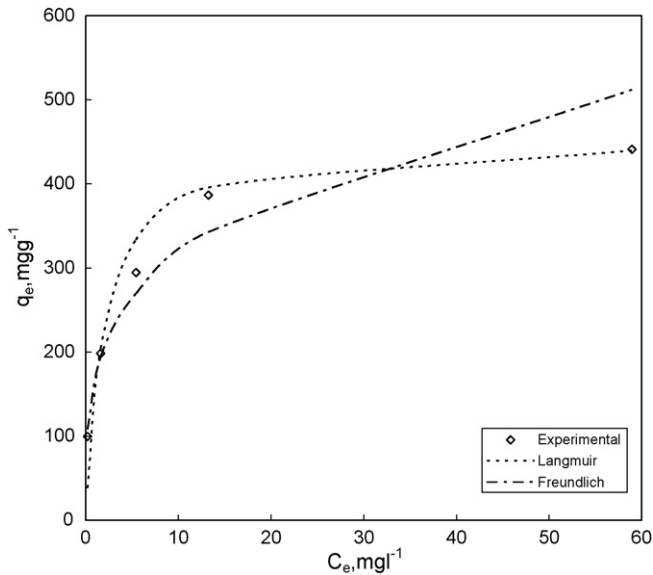
Fig. 2. Equilibrium adsorption isotherm of methylene blue onto activated carbon at 30 °C.

3.3.1. Langmuir isotherm

The linear form of Langmuir's isotherm model is given by the following equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \left(\frac{1}{Q_o}\right) C_e \qquad (3)$$

where C_e is the equilibrium concentration of the adsorbate (MB) (mg/l), q_e the amount of adsorbate adsorbed per unit mass of adsorbate (mg g^{-1}), and Q_o and b are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. When C_e/q_e was plotted against C_e, straight line with slope $1/Q_o$ was obtained (Fig. 3), indicating that the adsorption of MB on activated carbon follows the Langmuir isotherm. The Langmuir constants 'b' and 'Q_o' were calculated from this isotherm and their values are given in Table 1.
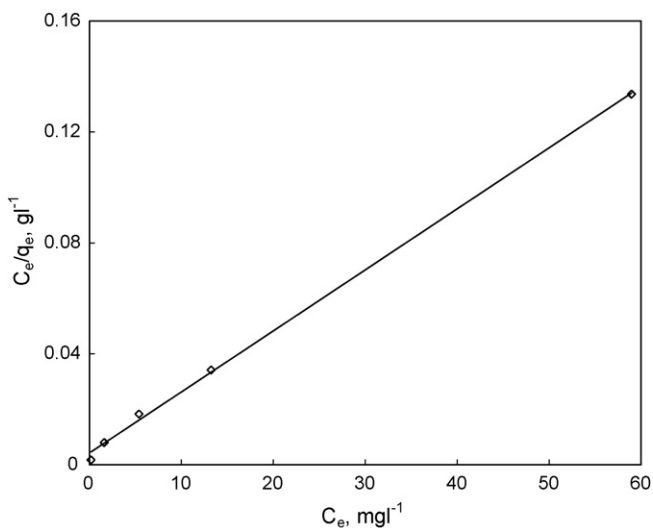

Fig. 3. Langmuir adsorption isotherm of methylene blue-activated carbon adsorption at 30 °C.

Table 1
Langmuir and Freundlich isotherm constants for MB at 30 °C

Langmuir isotherm	
Q_o (mg g^{-1})	454.20
b (mg^{-1})	0.518
R^2	0.999
R_L	4.0E−03
Freundlich isotherm	
$1/n$	0.268
K_F [(mg g^{-1})(mg^{-1})$^{1/n}$]	171.40
R^2	0.964

Conformation of the experimental data into Langmuir isotherm model indicates the homogeneous nature of bamboo carbon surface, i.e. each dye molecule/bamboo carbon adsorption has equal adsorption activation energy. The results also demonstrate the formation of monolayer coverage of dye molecule at the outer surface of bamboo carbon. Similar observation was reported by the adsorption of acid orange 10 dye onto activated carbons prepared from agricultural waste bagasse [6] and by the adsorption of direct dyes on activated carbon prepared from sawdust [19] and adsorption of Congo red dye on activated carbon from coir pith [20].

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter (R_L) [21], which is defined by:

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

where b is the Langmuir constant and C_0 the highest dye concentration (mg l^{-1}). The value of R_L indicates the type of the isotherm to be either 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 0.004 and confirmed that the activated carbon is favorable for adsorption of MB dye under conditions used in this study.

3.3.2. Freundlich isotherm

The well-known logarithmic form of Freundlich model is given by the following equation:

$$\log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e \qquad (5)$$

where q_e is the amount adsorbed at equilibrium (mg g^{-1}), C_e the equilibrium concentration of the adsorbate (MB) and K_F and n are Freundlich constants, n giving an indication of how favorable the adsorption process and K_F (mg g^{-1}(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. 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 [22]. A value for $1/n$ below one indicates a normal Langmuir isotherm while $1/n$ above one is indicative of cooperative adsorption [23]. The plot of $\log q_e$ versus $\log C_e$ gives straight lines with slope '$1/n$' (Fig. 4), which shows that the adsorption

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

Dyes	Adsorbent	Maximum monolayer adsorption capacity (mg g^{-1})	Reference
Methylene blue	Bamboo based activated carbon	454.20	This work
Methylene blue	Bamboo dust activated carbon	143.20	[27]
Methylene blue	Coconut shell activated carbon	277.90	[27]
Methylene blue	Groundnut shell activated carbon	164.90	[27]
Methylene blue	Rice husk activated carbon	343.50	[27]
Methylene blue	Straw activated carbon	472.10	[27]
Methylene blue	Jute fiber carbon	225.64	[6]
Basic red 46	Sludge-based activated carbon	188.00	[28]
Acid brown 283	Sludge-based activated carbon	20.50	[28]
Direct red 89	Sludge-based activated carbon	49.20	[28]
Direct black 168	Sludge-based activated carbon	28.90	[28]
Basic red 46	Chemviron GW activated carbon	106.00	[28]
Acid brown 283	Chemviron GW activated carbon	22.00	[28]
Direct red 89	Chemviron GW activated carbon	8.40	[28]
Direct black 168	Chemviron GW activated carbon	18.70	[28]
Congo red	Coir pith-based activated carbon	6.72	[20]

of MB also follows the Freundlich isotherm. Accordingly, Freundlich constants (K_F and n) were calculated and recorded in Table 1.

Table 1 shows the values of the parameters of the two isotherms and the related correlation coefficients. A comparison is also made between two isotherms plotted in Fig. 2, which shows the experimental data points and the two theoretical isotherms plotted on the same graph. As seen from Table 1, the Langmuir model yields a somewhat better fit ($R^2 = 0.999$) than the Freundlich model ($R^2 = 0.964$). As also illustrated in Table 1, the value of $1/n$ is 0.268, which indicates favorable adsorption [24].

Table 2 lists the comparison of maximum monolayer adsorption capacity of some dyes on various adsorbents. Compared with some data in the literature, Table 2 shows that the activated carbon studied in this work has very large adsorption capacity.
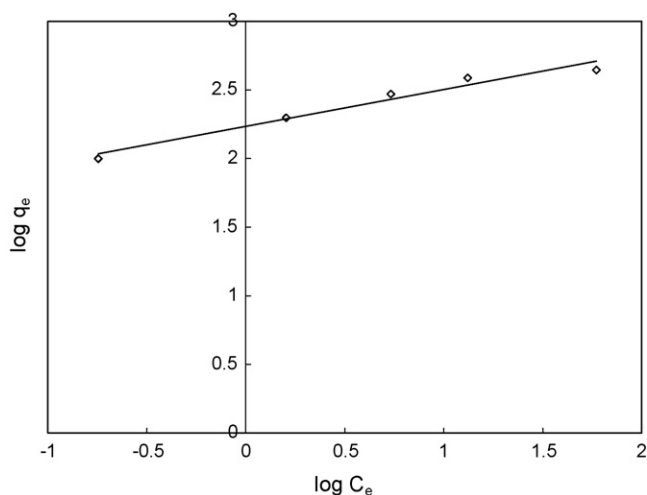
3.4. Adsorption kinetics

The rate constant of adsorption is determined from the pseudo first-order equation given by Langergren and Svenska [25]:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{6}$$

where q_e and q_t are the amounts of MB adsorbed (mg g^{-1}) at equilibrium and at time t (min), respectively, and k_1 the rate constant adsorption (h^{-1}). Values of k_1 were calculated from the plots of $\ln(q_e - q_t)$ versus t (Fig. 5) for different concentrations of MB. Although the correlation coefficient values at high concentration are higher than 0.80, the experimental q_e values do not agree with the calculated ones, obtained from the linear plots (Table 3). This shows that the adsorption of MB onto activated carbon is not a first-order kinetic.

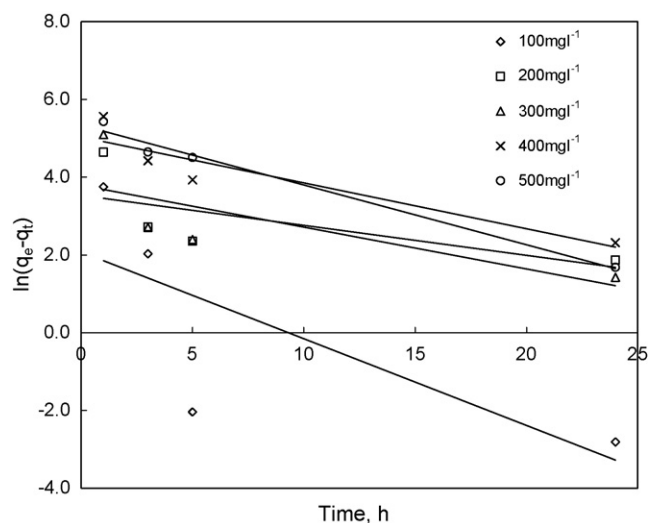Fig. 4. Freundlich adsorption isotherm of methylene blue-activated carbon adsorption at 30 °C.

Fig. 5. Pseudo-first-order kinetics for adsorption of methylene blue adsorption by activated carbon at 30 °C.

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

Initial concentration (mg^{-1})	$q_{e,exp}$ (mg g^{-1})	First-order kinetic model				Second-order kinetic model			
		k_1 (h^{-1})	$q_{e,cal}$ (mg g^{-1})	R^2	SSE (%)	k_2 [g (mg h)$^{-1}$]	$q_{e,cal}$ (mg g^{-1})	R^2	SSE (%)
100	99.8	0.2231	8.0	0.56	45.9	0.0286	100.0	0.99	0.1
200	198.4	0.0772	37.5	0.45	80.4	0.0076	200.0	0.99	0.8
300	294.6	0.1077	44.4	0.54	125.1	0.0058	294.1	0.99	0.7
400	386.8	0.1179	153.2	0.86	116.8	0.0018	400.0	0.99	5.9
500	441.1	0.1536	207.4	0.99	116.8	0.0019	454.5	0.99	6.0

On the other hand, a pseudo second-order equation based on equilibrium adsorption [26] is expressed as:

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_t}\right) t \tag{7}$$

where k_2 (g/mg h) is the rates constant of second-order adsorption. If second-order kinetics is applicable, the plot of t/q versus t should show a linear relationship. There is no need to know any parameter beforehand and q_e and k_2 can be determined from the slope and intercept of the plot. Also, this procedure is more likely to predict the behavior over the whole range of adsorption. The linear plots of t/q versus t (Fig. 6) show a good agreement between experimental and calculated q_e values (Table 3). The correlation coefficients for the second-order kinetic model are greater than 0.99 indicating the applicability of this kinetic equation and the second-order nature of the adsorption process of MB on activated carbon.

3.5. Test of kinetics models

Besides the value of R^2, the applicability of both kinetic models are verified through the sum of error squares (SSE, %). The adsorption kinetics of MB on AC was tested at different initial concentrations. The validity of each model was determined by

the sum or error squares (SSE, %) given by:

$$SSE (\%) = \sqrt{\frac{\sum (q_{e,exp} - q_{e,cal})^2}{N}} \tag{8}$$

where N is the number of data points.

The higher is the value of R^2 and the lower is the value of SSE, the better will be the goodness of fit. Table 3 lists the calculated results. It is found that the adsorption of methylene blue on AC can be best described by the second-order kinetic model. Similar phenomena processes have been observed in the adsorption of direct dyes on activated carbon prepared from sawdust [19] and adsorption of Congo red dye on activated carbon from coir pith [20].

4. Conclusions

The present investigation showed that bamboo can be effectively used as a raw material for the preparation of activated carbon for the removal of methylene blue dye from aqueous solution over a wide range of concentration. Methylene blue is found to adsorb strongly on the surface of activated carbon. Adsorption behaviour is described by a monolayer Langmuir type isotherm. Kinetic data follows pseudo second-order kinetic model. The value of the maximum adsorption capacity, Q_o, (454.2 mg g^{-1}) is comparable with the values for commercial activated carbon reported in earlier studies.

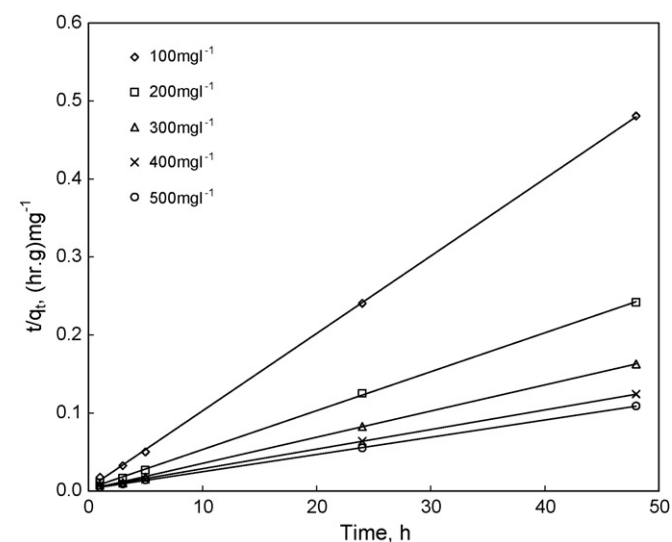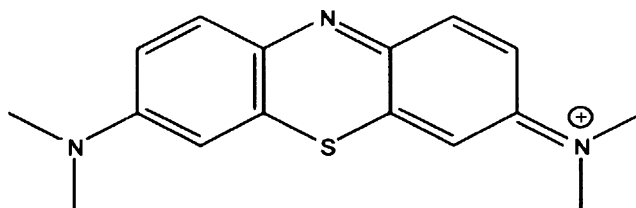### Appendix A. Chemical structure of methylene blue dye

Fig. 6. Pseudo-second-order kinetics for adsorption of methylene blue adsorption by activated carbon at 30 °C.

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