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Chemical Engineering Journal 127 (2007) 121–129

Chemical Engineering Journal

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Adsorption of basic dye onto activated carbon prepared from durian shell: Studies of adsorption equilibrium and kinetics

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Received 23 May 2006; received in revised form 9 September 2006; accepted 15 September 2006

Abstract

An activated carbon was prepared from durian shell and used for the removal of methylene blue from aqueous solutions. The activated carbon was prepared using chemical activation method with potassium hydroxide as the activating agent. The activation was conducted at 673.15 K for 1 h with mass ratio of chemical activating agent to durian shell 1:2. Batch kinetics and isotherm studies were conducted to evaluate the adsorption behavior of the activated carbon from durian shell. The adsorption experiments were carried out isothermally at three different temperatures. The Langmuir and Freundlich isotherm model were used to describe the equilibria data. The Langmuir model agrees with experimental data well. The Langmuir surface kinetics, pseudo first order and pseudo second order models were used to evaluate the kinetics data and the rate constant were also determined. The experimental data fitted very well with the Langmuir surface kinetics and pseudo first order model.

Keywords: Activated carbon; Adsorption; Durian shell

1. Introduction

Several industries, such as textile, ceramic, paper, printing and plastic use dyes in order to color their product. In the coloring process, these industries also consume substantial volumes of water, and as a result, large amount of colored wastewater are generated. The present of dyes in water is undesirable since even a very small amount of these coloring agents is highly visible and may be toxic to aquatic environment [1–5]. Several methods are available for color removal from wastewater such as membrane separation, aerobic and anaerobic degradation using various microorganisms, chemical oxidation, coagulation and flocculation, adsorption using different kind of adsorbents and reverse osmosis [1,3,5]. Among them, adsorption is a promising removal technique that produces effluents containing very low levels of dissolved organic compounds.

Considerable research has been conducted into the removal of dyes from water effluent using adsorption technique using different adsorbents such as activated carbon [2–5], fly ash [6], sawdust [7–9], corncob [10], barley husk [10], orange peel [11], dead or living biomass [12–17] and other low-cost adsorbents [1]. The most widely used adsorbent for this purpose is activated carbon, but commercially available activated carbons are expensive and so they may not be economical for wastewater treatment. Other untreated low-cost adsorbents often have low adsorption capacities; therefore their removal efficiencies are poor. If an activated carbon with high adsorption capacity for wastewater treatment purpose can be produced from low-cost or waste materials, then its use as an adsorbent should be economical.

Activated carbon is the most popular adsorbent for the adsorption process since it has high adsorption capacity. The adsorption capacity of activated carbon depends not only on its surface area, but also on its internal pore structure, surface characteristic and the presence of functional group on pore surface. Internal pore structure and surface characteristic play an important role in adsorption processes and depend both on the precursor used and method of preparation [18]. Different methods are available for characterizing the pore structure (surface area, pore volume, pore size distribution, etc.) of activated carbon such as small angle X-ray, mercury porosimetry, scanning electron microscopy and gas as well as liquid phase adsorption [19]. The characteristics of adsorption behavior of activated carbon are generally inferred in terms of both adsorption kinetics and equilibrium isotherm [20]. Therefore, to study both the adsorption kinetics and equilibrium, it is important to understand the adsorption mechanism

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doi:10.1016/j.cej.2006.09.011

Nomenclature

C_e	equilibrium concentration (mg/L)
C_o	initial concentration (mg/L)
C_t	solute concentration at the liquid phase at any time t (mg/L)
E_a	activation energy of adsorption (kJ/mol)
E_d	activation energy of desorption (kJ/mol)
k_a	the rate constant of adsorption (g/mg min)
k_d	desorption constant (min^{-1})
k_{do}	desorption rate constant at infinite temperature (min^{-1})
k_1	the rate constant for the first order (min^{-1})
k_2	rate constant of pseudo second order (g/mg min)
K_f	Freundlich adsorption constant (mg/g)
K_L	Langmuir equilibrium constant (L/mg)
K_o	adsorption equilibrium coefficient
m	mass of activated carbon (g)
n	parameter characterizes the heterogeneity of the system
q_e	equilibrium adsorption capacity (mg/g)
q_o	maximum adsorption capacity (mg/g)
q_t	the amount of solute adsorbed on the activated carbon at any time t (mg/g)
R	gas constant equal to 8.314 J/mol K
R_a	rate of adsorption
R_d	rate of desorption
t	time (min)
T	temperature (K)
V	volume of dye solution (L)

for the theoretical evaluation and interpretation of thermodynamic parameters [20–22].

The preparation of activated carbon can be carried out in two different processes: physical activation and chemical activation. Physical activation involves carbonization of carbonaceous materials followed by activation of the resulting char using gas activating agents, while in chemical activation both of carbonization and activation takes place in the same process in the presence of chemical agents. The chemical activation takes place at a temperature lower than that used in physical activation, therefore it can improve the pore development in the carbon structure because of the effect of chemicals. Furthermore, the carbon yields of chemical activation are higher than physical one [23].

Durian is one of the famous fruit commodities in Indonesia. The durian production every year is 600,000 tonnes, and the amount of durian shell generated approximately 350,000 tonnes, and direct discharge of this solid wastes will cause the environmental problems. Although there are many studies in the literature concerning to the preparation of activated carbon and its application for liquid phase adsorption, there is no information for the production and utilization of activated carbon from durian shell for color removal.

In this article, we report our study of the preparation of activated carbon from durian shell and the adsorption equilibrium and kinetics of methylene blue on this carbon. We also compared the applicability several well-known adsorption isotherm and kinetics models to describe the equilibria and dynamic.

2. Experimental technique

2.1. Preparation of activated carbon

Durian shells collected from local fruit stores in Surabaya were repeatedly washed with distilled water to remove dirt and other impurities and then dried at 393.15 K for 24 h to reduce the moisture content. The durian shell was grounded in micro hammer mill JANKE & KUNKEL. The proximate analysis of the precursor used in this study were 2.52, 5.53, 69.59 and 22.36% for moisture, ash, volatile matter and fixed carbon, respectively.

The preparation of activated carbon from durian shell was performed by chemical activation. Potassium hydroxide was used as chemical activating agent. The procedure of preparation of activated carbon using chemical activation method is as follows: 25 g of dried durian shell was mixed with 100 mL KOH solution, and then stirred at 303.15 K for about 5 h. The amount of KOH in the solution was adjusted to give mass ratio of chemical activating agent to durian shell 1:2. The resulting homogeneous slurry was dried at 383.15 K for at least 24 h.

The resulting samples were placed in a horizontal tubular reactor and then heated (at heating rate (10 K min^{-1}) at a carbonization temperature of 673.15 K. The carbonization and activation was performed under nitrogen flow of 150 cm^3 min^{-1} STP. Since carbonization time does not have much effect on the pore characteristic of activated carbon product [23,24], samples were held at final temperature for 1 h before cooling down under nitrogen flow. The activated carbon products were washed sequentially with a 0.5N HCl solution. Subsequently, the samples were repeatedly washed with hot distilled water until the pH of the solution reach 6.5 and finally washed with cold distilled water. After that, the samples were dried at 383.15 K for 24 h and stored in desiccator.

2.2. Activated carbon characterization

The pore structure characteristics of the resulting carbon were determined by nitrogen adsorption at 77.15 K using an automatic Micromeritics ASAP-2010 volumetric sorption analyzer. Prior to gas adsorption measurements, the carbon was degassed at 573.15 K in a vacuum condition for a period of at least 24 h. Nitrogen adsorption isotherms were measured over a relative pressure (P/P_o) range from approximately 10^{-5}–0.995. The Brunauer–Emmett–Teller (BET) surface area, micropore volume and micropore surface area of the activated carbons were determined by application of the Brunauer–Emmett–Teller and Dubinin–Asthakov (DA) analysis software available with the instrument, respectively. The BET surface area was determined by means of the standard BET equation applied in the relative pressure range from 0.06 to 0.3.

2.3. Adsorption procedure

Basic dye used in this study was methylene blue purchased from Sigma–Aldrich and it was used as received without further purification. The maximum wavelength of this dye is 663. Stock solutions were prepared by dissolving accurately 0.250 g of methylene blue in 1 L of distilled water. To prevent decolorization by direct sunlight, the stock solutions were stored in dark bottle and kept in dark place before being used.

Adsorption equilibrium and kinetics studies were conducted using static technique. Equilibrium data were obtained by adding 0.1–1.1 g of activated carbons into a series of 250 mL conical flasks each filled with 100 mL of dye solution with initial concentration of 250 mg/L. The conical flasks then covered with aluminum foil and were then placed in a thermostatic shaker bath (Memmert Type WB-14 equipped with an SV 1422 temperature controller) and shaken at 120 rpm for 96 h. During the adsorption the temperatures of system were kept constant at three different temperatures (303.15, 313.15 and 323.15 K). Product analysis showed that equilibrium conditions were reached after 96 h.

After equilibrium time had reached, the solutions were centrifuged (MLW T.51.1) at 2500 rpm for 5 min, and the clarified supernatant solutions were carefully decanted to be analyzed using a UV/VIS spectrophotometer (Shimadzu UV-1201). If the reading of absorbance in the spectrophotometer exceeded 0.7, the dye solutions were diluted. The final concentration of the solution was then determined from calibration curve.

The amount of dye adsorbed is calculated based on the following equation

$$q_e = \frac{C_o - C_e}{m} V \tag{1}$$

where q_e is the amount of dye adsorbed in activated carbon, C_o and C_e the initial and equilibrium concentration of dye solutions, respectively, m the amount of adsorbent and V is the volume of solution.

In the adsorption experiments the dye solutions were prepared by mixing weighed amount of dye with distilled water to yield concentration 200, 250 and 300 mg/L. The solutions were made up in a series of 0.25 L of conical flasks. The experiments were conducted in thermostatic shaker bath operating at 303.15, 313.15 and 323.15 K and 120 rpm. Prior the addition of carbon, the flasks containing 100 mL of dye solutions were placed in thermostatic bath for 30 min so that they could heat up to the operating temperature of experiment. At different time intervals a sample was taken from a flask (1 mL using micropipette). All samples were diluted with distilled water and analyzed immediately.

Here we also compare the adsorption capacity of activated carbon produced from durian shell with Filtrasorb-400 (F-400), a commercially available coal based activated carbon, produced by Calgon Carbon.

Table 1
Pore characteristics of DSAC and F-400

Pore characteristic	DSAC	F-400
BET surface area (m^2/g)	991.82	877.82
Micropore surface area (m^2/g)	849.31	761.80
Micropore volume (cm^3/g)	0.368	0.343
Total pore volume (cm^3/g)	0.471	0.468

3. Results and discussions

3.1. Pore characteristic of activated carbon

The pore characteristic of the activated carbon prepared from durian shell with chemical activation (DSAC) and Filtrasorb-400 are given in Table 1. The nitrogen adsorption of DSAC and F-400 are depicted in Fig. 1. From pore characteristic (given in Table 1) and nitrogen adsorption isotherm (Fig. 1) it can be seen that the activated carbon from durian shell (DSAC) has higher capacity than F-400, indicating that DSAC is promising candidate for adsorption application. The nitrogen gas adsorption isotherm of DSAC clearly shows that the nature of carbon is a combination of microporous and mesoporous.

The internal structures of microporous and mesoporous carbons are usually characterized in terms of the pore size distribution. The pore size distribution is a function of the assumed shape of the model pores used in the analysis. Here we used the DFT model to interpret the pore size distribution based on nitrogen adsorption. Fig. 2 shows that the DSAC has a microporous and mesoporous structure. The presence of mesopores together with micropores in the activated carbons enhances their adsorption capacities, especially for large molecules of adsorbates such as dye molecules [3].

3.2. Adsorption equilibrium studies

The analysis and design of adsorption separation processes requires the relevant adsorption equilibria, which is the most important piece of information in understanding an adsorption process [25]. The adsorption equilibrium data of methylene blue

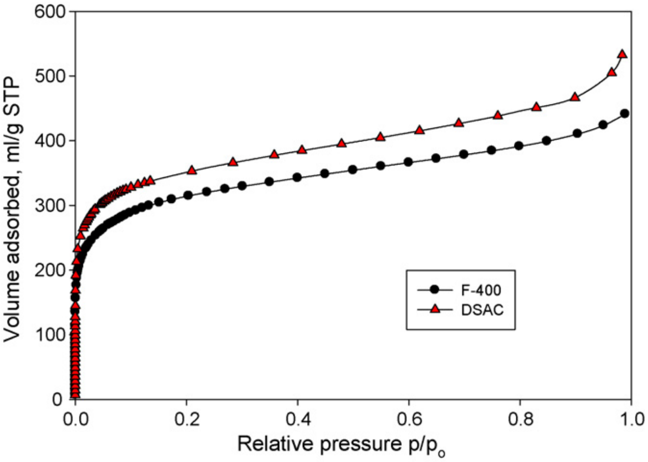

Fig. 1. Nitrogen adsorption isotherm of F-400 and DSAC.

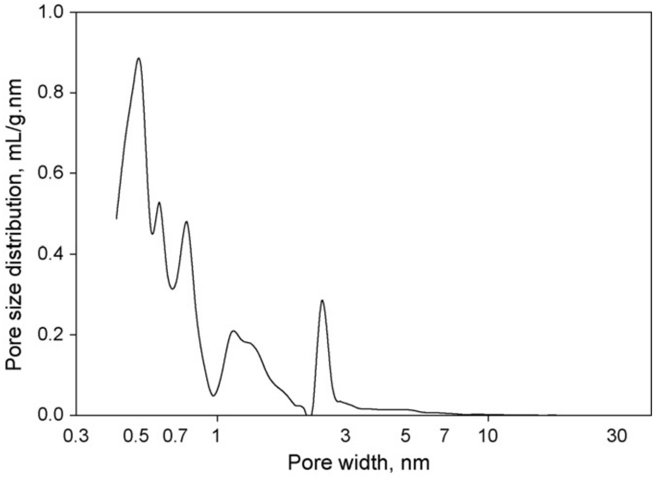

Fig. 2. Pore size distribution of DSAC.

on activated carbon derived from durian shell was fitted by several well-known isotherm models to assess their utility. These include Langmuir and Freundlich models.

Langmuir model is the most widely used isotherm equation, which has the form as follows

$$q_e = q_o \frac{K_L C_e}{1 + K_L C_e} \tag{2}$$

where q_o and K_L are Langmuir isotherm parameters, representing the maximum adsorption capacity for the solid phase loading and the Langmuir equilibrium constant related to the heat of adsorption, respectively. Fig. 3 depicts the adsorption isotherm of methylene blue on activated carbon from durian shell. In this figure, the experimental data are represented as symbols and the Langmuir model as solid lines. This figure clearly shows that the Langmuir equation can describe the experimental data fairly well. The optimal parameters from the fitting of Langmuir equation with the experimental data are given in Table 2. Temperature is well known to play an important role in adsorption in activated carbons, generally having a negative influence on the amount adsorbed [25]. The adsorption of organic compounds (including dye) is an exothermic process and the physical bonding between the organic compounds and the active sites of the carbon will weaken with increasing temperature. Also with the increase of temperature, the solubility of methylene blue also increases, the interaction forces between the solute and the solvent become stronger than solute and adsorbent, consequently the solute is more difficult to adsorb. Both of these features are consistent with the order of Langmuir adsorption capacity as seen in Table 2.

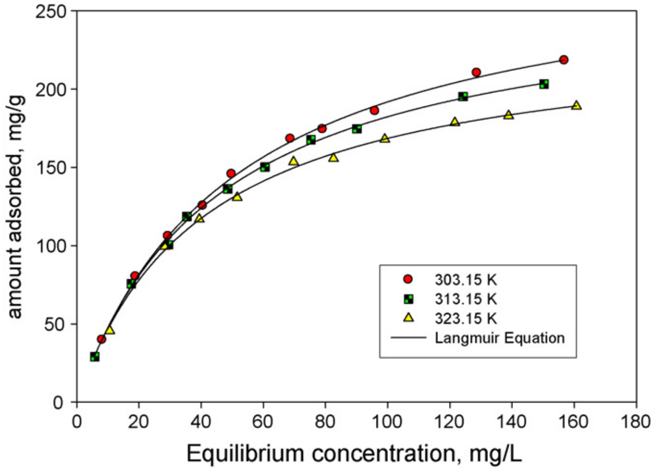

Fig. 3. Adsorption isotherm of methylene blue on DSAC and fits of Langmuir model.

Table 2
Parameters of Langmuir equation

Temperature (K)	q_o (mg/g)	K_L (L/mg)	R^2
303.15	289.26	0.0197	0.998
313.15	265.72	0.0219	0.998
323.15	237.13	0.0245	0.997

The heat of adsorption can be estimated from integrated van't Hoff equation, which relates the Langmuir equilibrium constant K to the temperature

$$K_L = K_o \exp\left[\frac{-E_a}{RT}\right] \tag{3}$$

here E_a is the activation energy of adsorption and K_o is the adsorption equilibrium coefficient. The gas constant R is equal to 8.314 J/mol K and T is the temperature of the solution. The magnitude of activation energy gives a type of adsorption, which is mainly physical or chemical. The range of 5–40 kJ/mol of activation energies correspond a physisorption mechanism or the range of 40–800 kJ/mol suggests a chemisorption mechanism [20]. The relation between the Langmuir equilibrium constant and $1/T$ is given in Fig. 4. The values of K_o and E_a obtained from Eq. (3) are 0.6883 L/g and 8.967 kJ/mol, respectively. The value of E_a obtained in this study indicating that the adsorption has low potential barrier and assigned to a physisorption.

The Freundlich isotherm is an empirical equation that is also often used to correlate adsorption experimental data. The Freundlich isotherm equation has the following form

$$q_e = K_f C_e^{1/n} \tag{4}$$

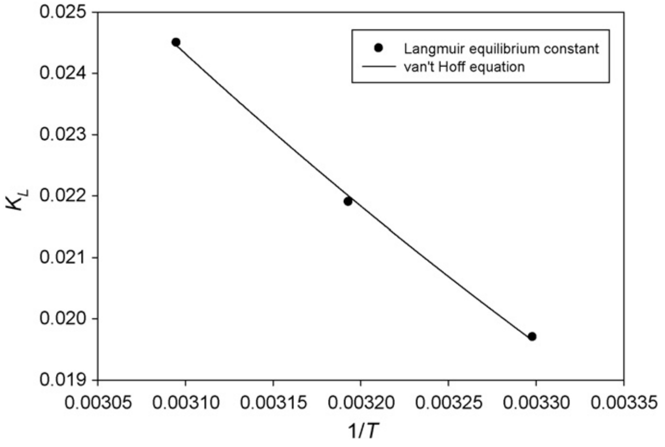

Fig. 4. Plot K_L vs. $1/T$ for methylene blue.

Table 3
Parameters of Freundlich equation

Temperature (K)	K_f (mg/g)	n	R^2
303.15	21.85	2.1422	0.973
313.15	21.93	2.1910	0.968
323.15	25.27	2.4601	0.958

where K_f is a parameter related to the adsorption capacity and parameter n characterizes the heterogeneity of the system. The parameters of Freundlich equation for system studied are given in Table 3, while the adsorption isotherm and model fit by Freundlich equation is depicted in Fig. 5. From this figure it is obvious that Freundlich equation fails to represent the adsorption data at low (because this equation does not incorporates Henry's law) and high concentration.

3.3. Adsorption kinetics studies

The studies of adsorption equilibria are important in determining the effectives of adsorption; however, it is also necessary to identify the types of adsorption mechanism in a given system. In this study we used three different models to predict the adsorption kinetic of methylene blue on activated carbon prepared from shell.

An approach to modeling both equilibrium and kinetics of adsorption is the Langmuir surface kinetics approach. The assumptions of this approach [26] are:

- Surface is homogeneous, that is adsorption energy is constant over all sites;
- adsorption on surface is localized, that is adsorbed atoms or molecules are adsorbed at definite, localized sites;
- each site can accommodate only one molecule or atom.

Based on this approach, the rate of adsorption, R_a, can be defined as

$$R_a = k_a C_t (q_e - q_t) \tag{5}$$

where k_a is the rate constant of adsorption, C_t the solute concentration at the liquid phase at any time t and q_t the amount of solute adsorbed on the activated carbon at any time t. The rate of desorption of solute, R_d, from the adsorbent surface to liquid phase can be written as

$$R_d = k_d q_t \tag{6}$$

where k_d is the desorption constant. Therefore, the rate of change of amount adsorbed at any time

$$\frac{dq_t}{dt} = R_a - R_d = k_a C_t (q_e - q_t) - k_d q_t \tag{7}$$

At equilibrium this equation become a well-known Langmuir equation (Eq. (2)). Rearrangement of Eq. (7) by combining the equilibrium condition (Eq. (2)) gives

$$\frac{dq_t}{dt} = \frac{k_a(1 + K_L C_e)(q_e - q_t)}{K} \tag{8}$$

since

$$K_L = \frac{k_a}{k_d} \tag{9}$$

Therefore, Eq. (8) can be written as

$$\frac{dq_t}{dt} = k_d(1 + K_L C_e)(q_e - q_t) \tag{10}$$

Integrating Eq. (10) yields

$$q_t = q_e(1 - \exp(-k_d(1 + K_L C_e)t)) \tag{11}$$

Eqs. (2) and (11) were used to model the kinetics of the adsorption process, and parameter k_d and q_e obtained at different initial concentrations and temperatures are summarized in Table 4. Figs. 6–8 depict the applicability of this model on the prediction of adsorption kinetics of methylene blue on durian shell activated carbon at different initial concentrations and temperatures. From Figs. 6–8 it is obvious that the Langmuir surface kinetics model can represent the data well. Also the fitted equilibrium adsorption capacities are in agreement with those experimental data as indicated in Table 4.

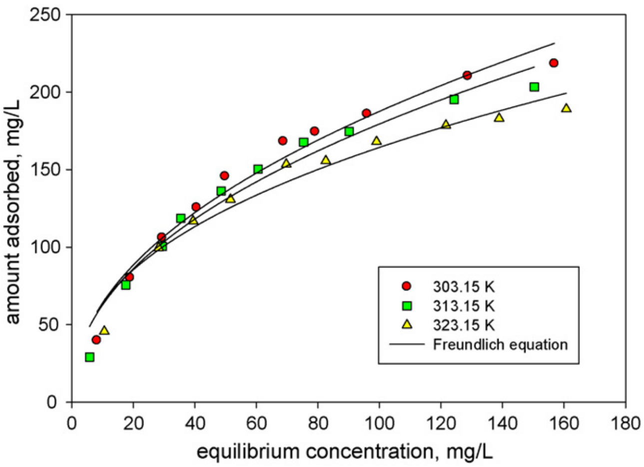

Fig. 5. Adsorption isotherm of methylene blue on DSAC and fits of Freundlich model.

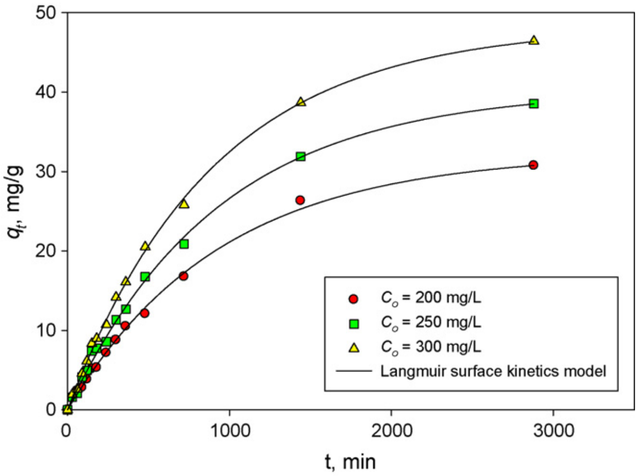

Fig. 6. Langmuir surface kinetic model for the adsorption of methylene blue on DSAC at 303.15 K.

Table 4
Fitted parameters values using Langmuir surface kinetics model

C_o (mg/L)	303.15 K			313.15 K			323.15 K		
	$k_d \times 10^3$ (min^{-1})	q_e (mg/g)	q_e (exp) (mg/g)	$k_d \times 10^3$ (min^{-1})	q_e (mg/g)	q_e (exp) (mg/g)	$k_d \times 10^3$ (min^{-1})	q_e (mg/g)	q_e (exp) (mg/g)
200	0.9204	32.74	32.27	1.419	32.48	32.28	1.974	32.31	32.26
250	0.9315	40.25	41.30	1.456	40.64	40.31	1.973	40.28	42.27
300	0.9314	48.24	47.30	1.461	48.33	48.31	1.989	47.88	49.05

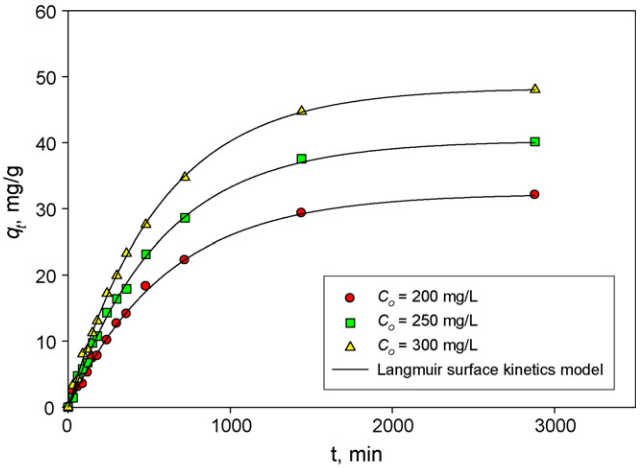

Fig. 7. Langmuir surface kinetic model for the adsorption of methylene blue on DSAC at 313.15 K.

The desorption energy can be calculated also from integrated van’t Hoff equation

$$k_d = k_{do}\ \exp\left[\frac{-E_d}{RT}\right] \tag{12}$$

where k_{do} and E_d are desorption rate constant at infinite temperature and activation energy of desorption, respectively. The energy of desorption can be obtained by plotting k_d versus $1/T$ as shown in Fig. 9. The value of k_{do} and E_d obtained by Eq. (12) are 107.68 min^{-1} and 29.28 kJ/mol, respectively.

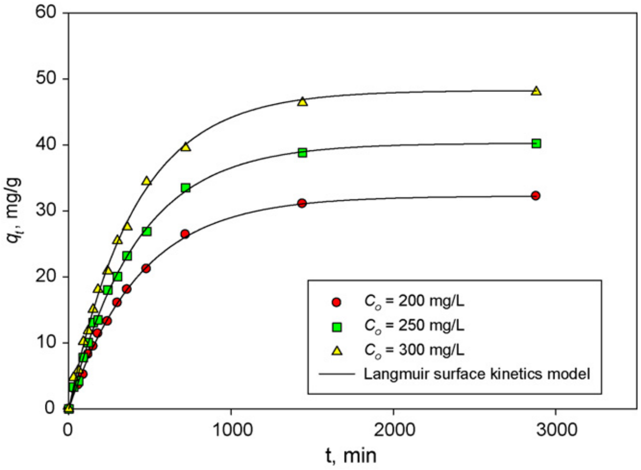

Fig. 8. Langmuir surface kinetic model for the adsorption of methylene blue on DSAC at 323.15 K.

Even this model can predict the adsorption kinetics data well as seen in Figs. 6–8, the energy of desorption (E_d) is larger than the heat of adsorption (E_a) that was calculated from the equilibrium data to be 8.967 kJ/mol. However, both of adsorption and desorption energies still correspond a physisorption mechanism. This discrepancy could be due to the fact that this model has ignored the energetic heterogeneity of the carbon surface and the distribution of pore sizes.

Another model widely used to describe the adsorption kinetics is pseudo first order model which is also known as Lagergren equation. This equation has the form as follows

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{13}$$

where k_1 is the rate constant for the first order (min^{-1}). Integrating Eq. (13) for the boundary conditions $t = 0$–t and $q_t = 0$–q_t gives

$$q_t = q_e(1 - \exp(-k_1 t)) \tag{14}$$

The rate constant for the pseudo first order model (k_1) and equilibrium adsorption capacity (q_e) were determined using Sigma Plot software. The first order kinetics constant for the adsorption of methylene blue on DSAC at different initial concentrations and temperatures is given in Table 5. For the adsorption of methylene blue on DSAC, the pseudo first order kinetics is applicable for the system studied as shown in Figs. 10–12, furthermore the value of q_e obtained from the plot also agree with experimental values. Similar result was also obtained by Garg et al. for removal methylene blue using Indian Rosewood sawdust [27].

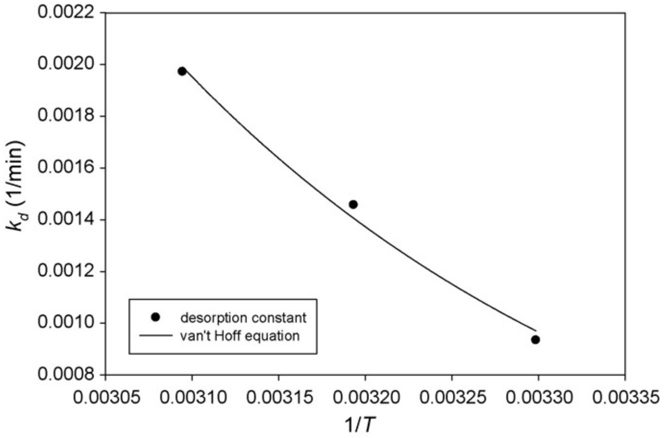

Fig. 9. Determination of energy of desorption.

Table 5
Fitted parameters values using pseudo first order kinetics model

C_o (mg/L)	303.15 K			313.15 K			323.15 K		
	$k_1 \times 10^3$ (min^{-1})	q_e (mg/g)	q_e (exp) (mg/g)	$k_1 \times 10^3$ (min^{-1})	q_e (mg/g)	q_e (exp) (mg/g)	$k_1 \times 10^3$ (min^{-1})	q_e (mg/g)	q_e (exp) (mg/g)
200	1.036	32.74	32.27	1.615	32.48	32.28	2.284	32.30	32.26
250	1.082	40.25	41.30	1.716	40.64	40.31	2.377	40.28	42.27
300	1.118	48.24	47.30	1.785	48.33	48.31	2.498	47.87	49.05

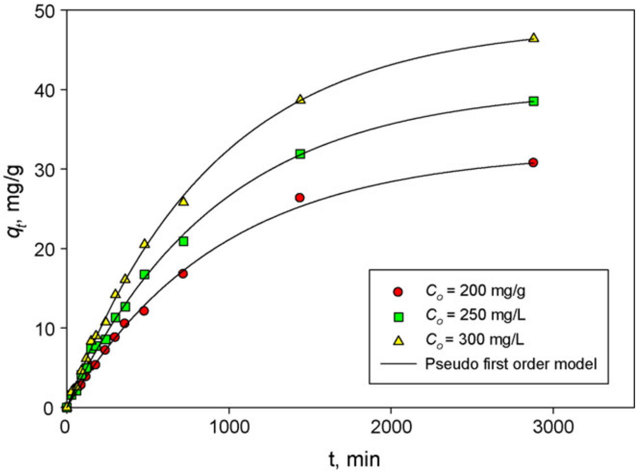

Fig. 10. Pseudo first order model for the adsorption of methylene blue on DSAC at 303.15 K.

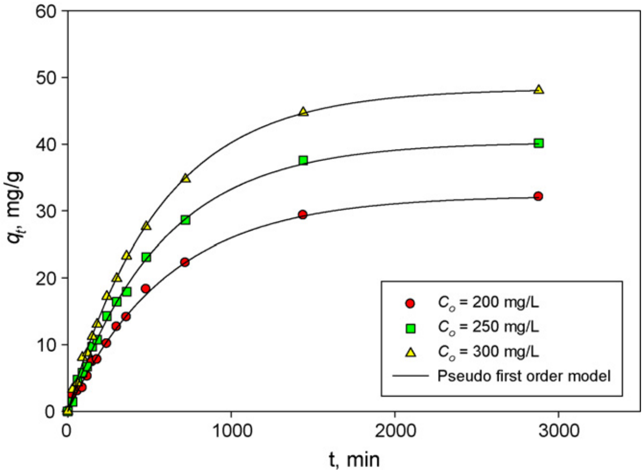

Fig. 11. Pseudo first order model for the adsorption of methylene blue on DSAC at 313.15 K.

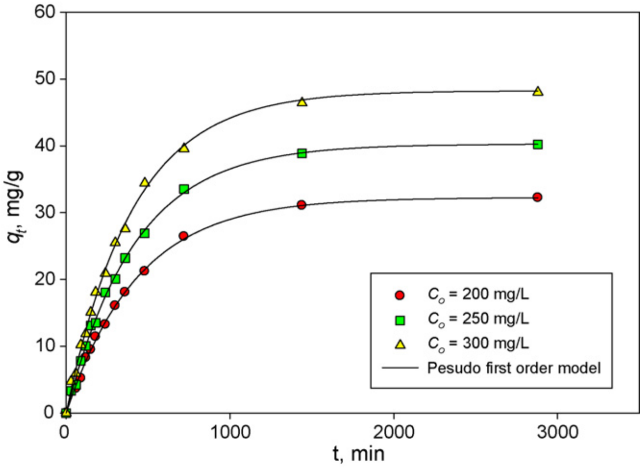

Fig. 12. Pseudo first order model for the adsorption of methylene blue on DSAC at 323.15 K.

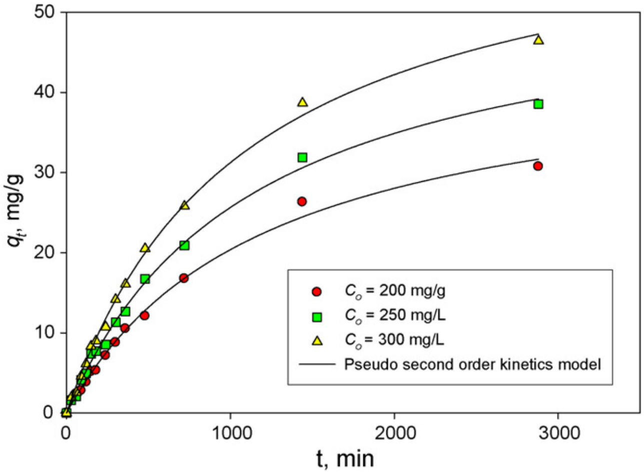

Fig. 13. Pseudo second order model for the adsorption of methylene blue on DSAC at 303.15 K.

Kinetics data were further treated with the pseudo second order kinetics model. The differential equation of this model is written as follows

$$\frac{\mathrm{d}q_t}{\mathrm{d}t} = k_2(q_e - q_t)^2 \qquad (15)$$

where k_2 is the rate constant of pseudo second order (g/mg min). Integrating Eq. (15) for the boundary condition $t = 0$–t and $q_t = 0$–q_t gives

$$q_t = q_e\left(\frac{q_e k_2 t}{1 + q_e k_2 t}\right) \qquad (16)$$

which is the integrated rate law for a pseudo second order reaction. In this model q_e and k_2 are the fitted parameters. The fitted parameter q_e and k_2 obtained from optimization using Sigma Plot software are listed in Table 6 and the model curves are plotted in Figs. 13–15. As can be seen in these figures that this model can adequately represent the adsorption kinetics data of methy-

Table 6
Fitted parameters values using pseudo second order kinetics model

C_o (mg/L)	303.15 K			313.15 K			323.15 K		
	$k_2 \times 10^5$ (g/mg min)	q_e (mg/g)	q_e (exp) (mg/g)	$k_2 \times 10^5$ (g/mg min)	q_e (mg/g)	q_e (exp) (mg/g)	$k_2 \times 10^5$ (g/mg min)	q_e (mg/g)	q_e (exp) (mg/g)
200	1.85	44.89	32.27	3.57	41.30	32.28	5.92	39.12	32.26
250	1.63	54.52	41.30	3.14	51.08	40.31	5.01	48.56	42.27
300	1.42	65.07	47.30	2.79	60.41	48.31	4.51	57.43	49.05

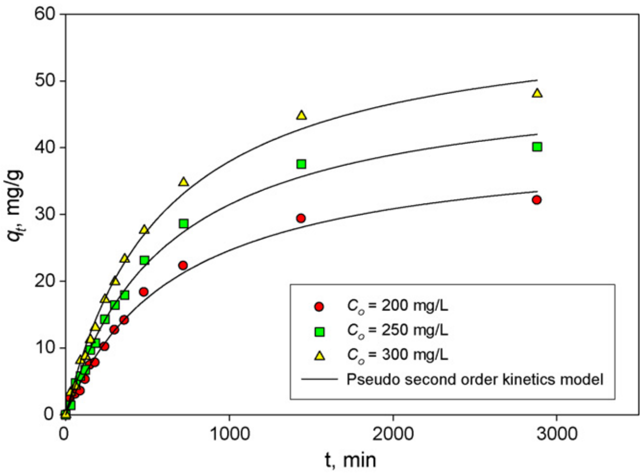

Fig. 14. Pseudo second order model for the adsorption of methylene blue on DSAC at 313.15 K.

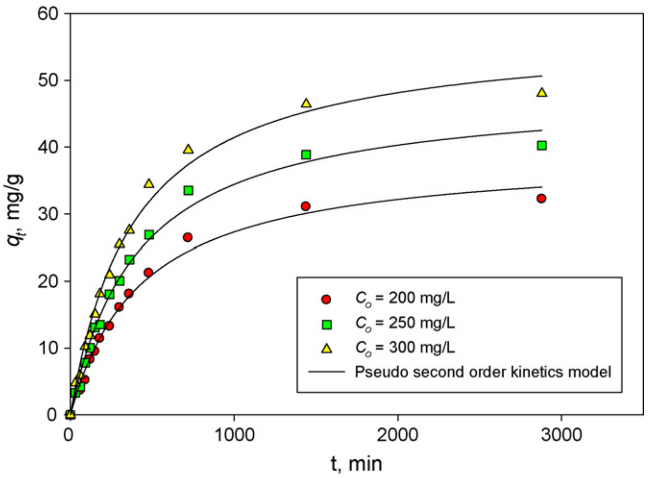

Fig. 15. Pseudo second order model for the adsorption of methylene blue on DSAC at 323.15 K.

lene blue on DSAC. However, the values of q_e from the pseudo second order kinetics are not in agreement with experimental data (see Table 6). This confirms that the chemisorption is not the controlling mechanism for the adsorption of methylene blue on activated carbon prepared from durian shell.

4. Conclusions

This study shows that the activated carbon prepared from durian shell with chemical activation is a potential adsorbent for methylene blue removal. The batch adsorption experiments were conducted isothermally at three different temperatures (303.15, 313.15 and 323.15 K). For the adsorption equilibrium, it was found that Langmuir model can represent the data well compared with Freundlich isotherm. The adsorption kinetics of methylene blue can be well described by both of the pseudo first model equation and Langmuir surface kinetics model.

Acknowledgements

This research was supported by Sub-Project Management Unit Technological and Professional Skills Development Sector Project (ADB Loan No. 1792-INO) through Widya Mandala TPSDP Student Grant 2006.

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