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Adsorption studies of basic dye on activated carbon derived from agricultural waste: *Hevea brasiliensis* seed coat

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Abstract

Activated carbon prepared from rubber (*Hevea brasiliensis*) seed coat was used to remove basic blue 3 (BB3) from aqueous solutions. Batch adsorption studies were conducted to evaluate the effect of contact time, and initial concentration (50–500 mg/L) on the removal of BB3 at temperature of 30 °C. The equilibrium adsorption data of BB3 on activated carbon were analyzed by the Langmuir, Freundlich and Temkin isotherm models. The isotherm data were well described by the Freundlich isotherm model. The monolayer adsorption capacity was 227.27 mg/g at 30 °C. Pseudo-first-order, pseudo-second-order and intraparticle diffusion models were used to analyze the kinetic data obtained at different concentrations. The adsorption kinetics was well described by the pseudo-second-order kinetic model. Rubber seed coat-based activated carbon was shown to be a promising material for adsorption of BB3 from aqueous solutions.

Keywords: Activated carbon; Rubber seed coat; Basic blue; Adsorption isotherm; Kinetics

1. Introduction

Textile and other industries in Malaysia used dyes and pigments to color their products. Dye pollutants from these industries are important sources of environmental contamination. The wastewater discharged from these industries may contain chemicals that exhibit toxic effects toward microbial populations and can be toxic and carcinogenic to animals. Basic dyes are water soluble cationic dyes that are mainly applied to acrylic fibres but find some use for wool and silk. Their tinctorial value is very high where less than 1 ppm of the dye produces obvious coloration [1]. Also these dyes may drastically affect photosynthetic phenomenon in aquatic life due to reduced light penetration [2,3]. As a result, the removal of color from waste effluents has become environmentally important. Various methods including coagulation [4], chemical oxidation [5], photocatalysis [6,7], electrochemical [8] and adsorption techniques have been examined. Among these methods, the adsorption onto activated carbon has been found to be superior compared to other techniques. However, commercially available activated carbons are still considered expensive [9]. This is due to the use of non-renewable and relatively expensive starting material such as coal, which is unjustified in pollution control applications [10]. Therefore, in recent years, this has prompted a growing research interest in the production of activated carbons from renewable and cheaper precursors which are mainly industrial and agricultural by-products, especially for application concerning wastewater treatment. Researchers have studied the production of activated carbons from rubber seed coat [11], pecan shells [12], jute fibre [13], Indian rosewood sawdust [1], olive stones [14], pinewood [15], sawdust [16], coir pith [17], rice husk [18], bamboo [19], rattan sawdust [20] and oil palm fibre [21].

Hevea brasiliensis or known commonly as rubber tree is the main source of natural rubber. The tree is cultivated in large commercial scale in several countries in the tropics amounting to 9.485 million ha worldwide [22]. Apart from its latex, rubber tree has also been harnessed for its wood for making furniture and the seeds for para rubber seed oil used for manufacturing soap, paint, varnishes, fertilizer and animal feeds [23]. Rubber seed coat, a waste agricultural by-product, is utilized in Malaysia as fuel and manure. To produce a value added product from rubber seed coat, it is proposed to convert it to activated carbon. Rengaraj et al. [11] reported that activated carbon prepared from rubber seed coat is 2.25 times more efficient compared to commercial activated carbon for phenolic wastewater treatment. Hence, the

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feasibility of applying activated carbon from rubber seed coat towards the removal of basic dye from aqueous solution was approached.

The focus of the research was to evaluate the adsorption potential of rubber seed coat-based activated carbon for basic dye (BB3). The kinetic data and equilibrium data of adsorption studies were processed to understand the adsorption mechanism of basic blue 3 onto the prepared activated carbon.

2. Materials and methods

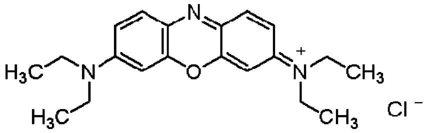
2.1. Adsorbate: basic blue 3

Basic blue 3 (BB3) was purchased from Sigma–Aldrich (M) Sdn Bhd, Malaysia and used without purification. The characteristics of the dye are listed in Table 1.

2.2. Preparation and characterization of activated carbon

The rubber (*H. brasiliensis*) seed coat was collected from a rubber plantation estate in Tanah Merah, Kelantan, Malaysia and was used as the precursor for the preparation of activated carbon in this work. The precursor was first washed with water to remove dirt. It was then dried and ground to particle size of −6 + 40 mesh. The precursor was carbonized at 700 °C under purified nitrogen (99.995%) flow of 150 cm^3/min for 2 h in a stainless steel vertical tubular reactor placed in a tube furnace. The char produced was then added with potassium hydroxide pallets, with KOH:char ratio (dry weight of KOH pallets (g)/dry weight of char (g)) of 1:1. Distilled water was then added to dissolve all the KOH pallets. After that, the mixture was dehydrated in an oven overnight at 105 °C. Then, it was activated to a final temperature of 850 °C. Once the final temperature was reached, the nitrogen gas flow was switched to carbon dioxide and activation was held for 2 h. The activated product was then cooled to room temperature and washed with hot deionized water and hydrochloric acid of 0.1 M until the pH of the washing solution reached 6–7 [21]. Then, the material was dried in an oven at 105 °C. Finally, the dried material was ground and sieved to get the particle size of 125–250 μm and stored in plastic containers for further use.

Table 1
Properties and characteristics of BB3

Generic name	Basic blue 3
Color index number	51004
Abbreviation	BB3
Commercial name	Cationic blue X-GB
Molecular formula	$C_{20}H_{26}ClN_3O$
Molecular weight (g/mol)	359.89
λ_{max} (nm)	654
Chemical structure	

The Brunauer–Emmett–Teller (BET) [24] was used for the characterization of the activated carbon. Surface morphology and the presence of porosity of the activated carbon prepared in this work were studied using scanning electron microscopy (SEM) analysis.

2.3. Batch equilibrium studies

Hundred milliliters of BB3 solutions with different initial concentrations (50–500 mg/L) were prepared in six conical flasks (250 mL). Equal masses of 0.1 g of activated carbon were added to BB3 solutions and kept in an isothermal shaker (30 ± 1 °C, 125 rpm) for 8 h to reach equilibrium of the solid–solution mixture. The flasks were then removed from the shaker and sample was withdrawn by syringe to be analyzed by a UV–vis spectrophotometer (Shidmadzu UV-1601, Japan) at 654 nm. The amount of adsorbate adsorbed at equilibrium, q_e (mg/g), was calculated by

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

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).

2.4. Batch kinetic studies

The kinetic experiments had a similar procedure with those of equilibrium tests. The samples were taken at present time intervals, and the concentrations of dye were similarly measured. The amount of adsorption at time t, q_t (mg/g), was calculated by

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

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

3. Results and discussion

3.1. Characterization of the prepared activated carbon

The BET surface area of the activated carbon derived from rubber seed coat prepared in this study was 1225 m^2/g with a total pore volume of 0.85 cm^3/g. The average pore diameter of the activated carbon was found to be 2.43 nm. The activated carbon contained relatively large surface area compared to commercially available activated carbons such as Darco activated carbon (20–40 mesh) (Aldrich), granulated activated carbon (Scharlau), no. 3 QP activated carbon (Panreac), 2.5 mm granular activated carbon (Merck) (Albus) and coconut shell activated carbon with surface area of 564, 1111, 800, 1184 and 774 m^2/g, respectively [25].

The SEM image of the prepared activated carbon is shown in Fig. 1. Many large pores were clearly found on the surface of the activated carbon. The well-developed pores had led to the large surface area and porous structure of the activated carbon.

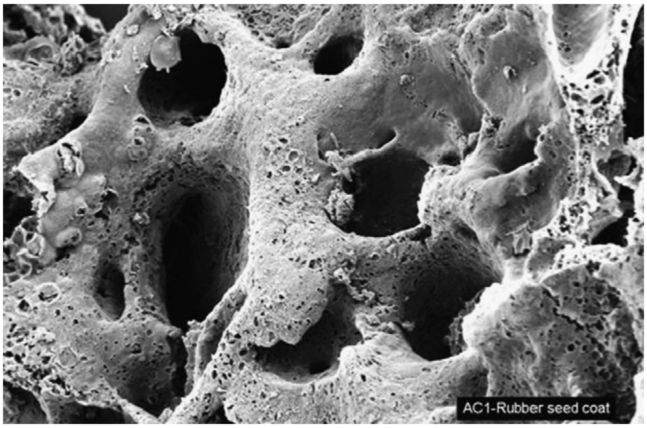


Fig. 1. Scanning electron micrograph of the prepared activated carbon (magnification = 2.00k ×).

3.2. *Effect of initial concentration and contact time on BB3 dye adsorption*

Fig. 2 shows the effect of initial concentration of BB3 on the adsorption of dye. At the beginning of adsorption, the adsorption amounts of BB3 were very high and saturations were reached after some time. The amount of dye adsorbed per unit mass of adsorbent increased with increase in dye concentration. When the initial concentrations increased from 50 to 500 mg/L, the amount of BB3 adsorbed at equilibrium (q_e) increased from 48.82 to 227.36 mg/g indicating that the initial BB3 concentration played an important role in the adsorption of BB3 on activated carbon. The initial concentration provides the necessary driving force to overcome the resistances to the mass transfer of BB3 between the aqueous and solid phases [26].

The adsorption of BB3 on activated carbon was also studied as a function of contact time in order to find out the equilibrium time for maximum adsorption. For initial concentration of 50–110 mg/L, the uptake of dye is very fast at the beginning and the equilibrium times were reached in 1 h. For higher concentration, the equilibrium times were reached in more than 5 h. The BB3 ions were adsorbed initially on the external surface area of activated carbon which made the adsorption rate very fast. When the adsorption of the exterior surface reached saturation, the BB3 ions entered into the pores of activated carbon and were adsorbed by the interior surface of the particles. This phenomenon took relatively long contact time. Similar phenomenon was reported in the literature for the adsorption of basic dye (methylene blue) on activated carbon prepared from rattan sawdust [20].

3.3. *Equilibrium modeling*

It is important to establish the most appropriate correlation for the equilibrium curves. An adsorption isotherm describes the relationship between the amount of adsorbate that is adsorbed on the adsorbent and the concentration of dissolved adsorbate in the liquid at equilibrium. Isotherm models such as Langmuir, Freundlich and Temkin have been used to describe the equilibrium nature of adsorption.

The Langmuir model [27] is the most frequently used model for the sorption of a pollutant from a liquid solution. This model is obtained under the ideal assumption of a totally homogenous adsorption surface. It is then assumed that once an adsorbate molecule occupies a site, no further adsorption can take place at that site. As a result, a saturation value is reached and no further adsorption can take place. The Langmuir model is given below:

$$q_e = \frac{q_{max} K_L C_e}{1 + K_L C_e} \qquad (3)$$

or in a linear form gives

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{1}{q_{max}} C_e \qquad (4)$$

where C_e is the equilibrium concentration (mg/L), q_e the amount of adsorbate adsorbed (mg/g), q_{max} the monolayer capacity (mg/g) and K_L is the adsorption equilibrium constant (L/mg). A plot of C_e/q_e versus C_e (Fig. 3) gives the adsorption coefficients. The q_{max} and K_L determined from the Langmuir isotherm were 227.27 mg/g and 0.044 L/mg, respectively, at 30 °C with R^2 of 0.97.

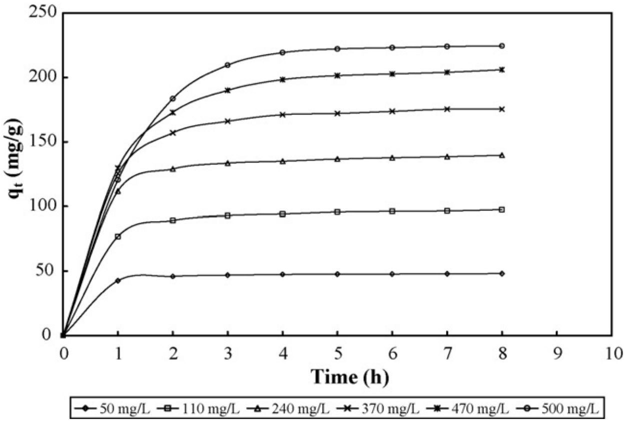


Fig. 2. Uptake of BB3 on prepared activated carbon vs. contact time at different initial concentrations at 30 °C (W = 0.1 g).

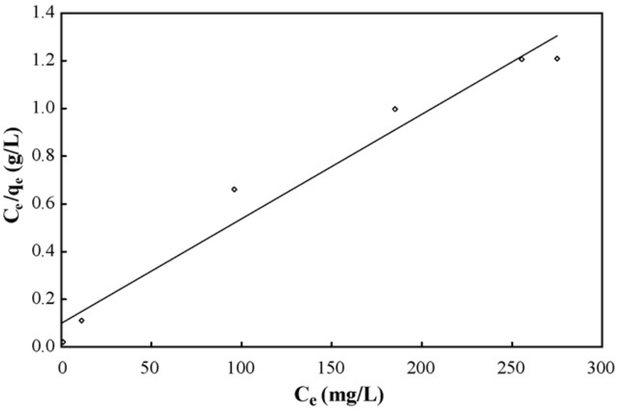


Fig. 3. Langmuir isotherm for adsorption of BB3 onto prepared activated carbon at 30 °C.

The essential characteristic of a Langmuir isotherm can be expressed in terms of a dimensionless separation factor, R_L [28]:

$$R_L = \frac{1}{1 + K_L C_0} \quad (5)$$

where K_L is the Langmuir constant and C_0 is the highest initial dye concentration (mg/L). If

$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

The value of R_L for adsorption of BB3 onto activated carbon was 0.102. This value indicated that the adsorption behaviour of activated carbon was favorable for the dye ($R_L < 1$).

The Freundlich isotherm is derived by assuming a heterogeneous surface with a non-uniform distribution of heat of adsorption over the surface [29]. This isotherm is suitable for a highly heterogeneous surface. The application of the Freundlich equation suggests that sorption energy exponentially decreases on completion of the sorptional centres of an adsorbent. The isotherm is expressed by the following equation:

$$q_e = K_F C_e^{1/n} \quad (6)$$

where K_F and n are the Freundlich constants that point to relative capacity and adsorption intensity, respectively. Linearization of Freundlich equation by taking logarithms gives

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (7)$$

Thus the constants can be determined as shown in Fig. 4. Values of K_F and n were determined as 49.97 and 3.87, respectively, at 30 °C with R^2 of 0.99. The value of n greater than unity indicated that BB3 was favorably adsorbed by activated carbon.

The Temkin equation [30] suggests a linear decrease of sorption energy as the degree of completion of the sorptional centres of an adsorbent is increased. The heat of adsorption of all the molecules in the layer would decrease linearly with coverage due to adsorbent–adsorbate interactions. The adsorption is characterized by a uniform distribution of binding energies, up to some maximum binding energy. The Temkin isotherm has been generally applied in the following form

$$q_e = \frac{RT}{b} \ln(A C_e) \quad (8)$$

and can be linearized as

$$q_e = B \ln A + B \ln C_e \quad (9)$$

where $B = RT/b$, b is the Temkin constant related to heat of sorption (J/mol), A the Temkin isotherm constant (L/g), R the gas constant (8.314 J/(mol K)) and T is the absolute temperature (K). Therefore, by plotting q_e versus $\ln C_e$ enables one to determine the constants A and B as shown in Fig. 5. The constants A and B are listed in Table 2.

From Table 2, the R^2 for the Freundlich isotherm is significantly higher than the other isotherms. The order of R^2 was Temkin < Langmuir < Freundlich. Therefore, the Freundlich isotherm is the best fit for BB3 adsorption. A similar result was reported for the adsorption of basic yellow dye on granular activated carbon produced from coffee grounds (ACP) [31].

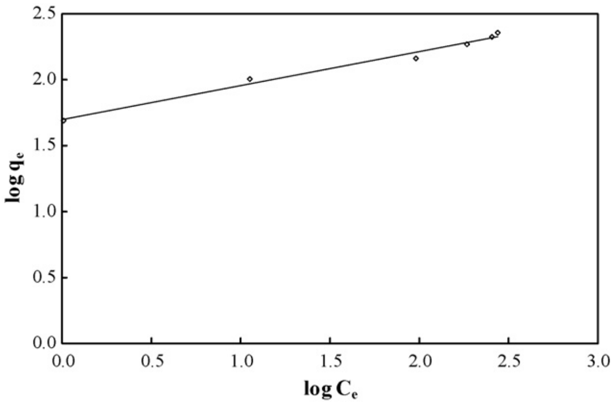


Fig. 4. Freundlich isotherm for adsorption of BB3 onto prepared activated carbon at 30 °C.

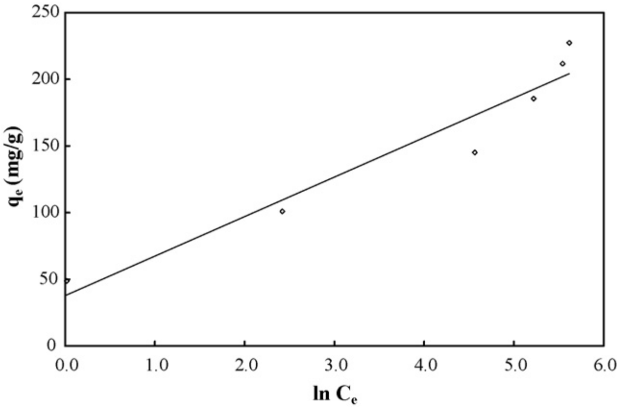


Fig. 5. Temkin isotherm for adsorption of BB3 onto prepared activated carbon at 30 °C.

Table 2
Langmuir, Freundlich and Temkin isotherm model constants and correlation coefficients for adsorption of BB3 onto prepared activated carbon at 30 °C

Isotherm	Parameters	
Langmuir	q_{max} (mg/g)	227.27
	K_L (L/mg)	0.044
	R^2	0.97
	R_L	0.102
Freundlich	K_F	49.97
	n	3.87
	R^2	0.99
Temkin	A	3.58
	B	85.02
	R^2	0.93

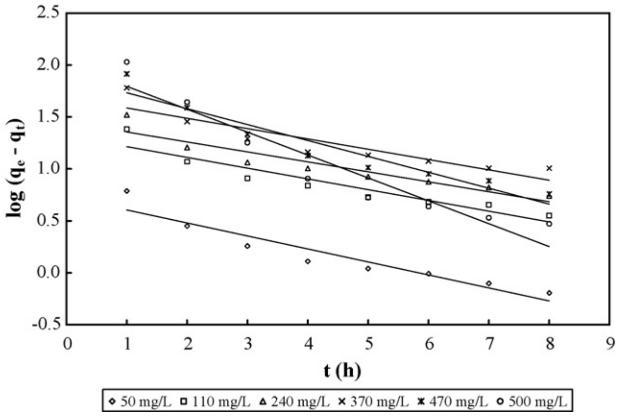


Fig. 6. Pseudo-first-order kinetic fit for adsorption of BB3 onto prepared activated carbon at 30 °C.

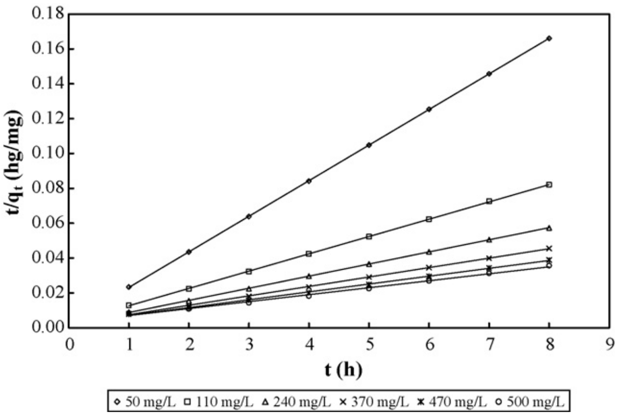


Fig. 7. Pseudo-second-order kinetic fit for adsorption of BB3 onto prepared activated carbon at 30 °C.

3.4. Kinetic modeling

Kinetic models are used to determine the rate of the adsorption process. Three kinetic models: pseudo-first-order, pseudo-second-order and intraparticle diffusion models were used to investigate the adsorption process of BB3 on activated carbon. Lagergren proposed a method for adsorption analysis which is the pseudo-first-order kinetic equation [32] in the form

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{10}$$

where q_e and q_t are the amounts of BB3 adsorbed at equilibrium and at time t in mg/g, respectively, and k_1 is the pseudo-first-order rate constant (h^{-1}).

The integration of Eq. (10) with the initial condition, $q_t = 0$ at $t = 0$ leads to

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \tag{11}$$

A linear plot of $\log(q_e - q_t)$ against time allows one to obtain the rate constant. If the plots were found to be linear with good correlation coefficient, indicating that Lagergren's equation is appropriate to BB3 adsorption on activated carbon. So, the adsorption process is a pseudo-first-order process [32,33]. The pseudo-first-order rate constants for this study were calculated from slopes of the plots (Fig. 6) of Eq. (11). The values of k_1 and calculated q_e were listed in Table 3.

The pseudo-second-order kinetic model of [34] can be represented in the following form

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

Plot of t/q_t versus t (Fig. 7) enables one to determine q_e and k_2 from the slope and intercept. The calculated R^2 for pseudo-second-order kinetic model are shown in Table 3.

From Table 3, the calculated R^2 are all unity for pseudo-second-order kinetic model. Therefore, the adsorption reaction can be most satisfactorily by the pseudo-second-order kinetic model for BB3 adsorption onto activated carbon found on the hypothesis that the rate limiting step may be chemisorption relating valency forces through sharing or exchange of electrons between adsorbent and adsorbate. The similar phenomenon has also been observed in the adsorption of basic blue 9 on activated carbon done by [35], the adsorption of methylene blue on activated carbons [36] and adsorption of bismark brown *R* on activated carbon prepared from rubberwood sawdust [37].

The transportation of adsorbate from solution phase to the surface of the adsorbent particles may be controlled either by one or more steps, e.g. film or external diffusion, pore diffusion, surface diffusion and adsorption on the pore surface, or a combination of more than one step. The possibility of intraparticle diffusion was explored by using the intraparticle diffusion model

Table 3
Pseudo-first and -second-order rate constants for adsorption of BB3 onto prepared activated carbon at different initial concentration at 30 °C

C_0 (mg/L)	Pseudo-first-order kinetic model				Pseudo-second-order kinetic model			
	k_1 (h^{-1})	$q_{e,cal.}$ (mg/g)	R^2	Δq_e (%)	k_2 (g/(mg h))	$q_{e,cal.}$ (mg/g)	R^2	Δq_e (%)
50	0.288	5.38	0.90	98.88	0.154	49.02	1.00	0.56
110	0.238	20.77	0.89	92.74	0.036	101.01	1.00	1.27
240	0.221	28.22	0.88	94.02	0.027	142.86	1.00	1.16
370	0.229	48.46	0.84	88.89	0.014	185.19	1.00	2.66
470	0.353	76.86	0.92	76.32	0.008	222.22	1.00	3.84
500	0.507	103.35	0.96	63.39	0.005	250.00	1.00	6.65

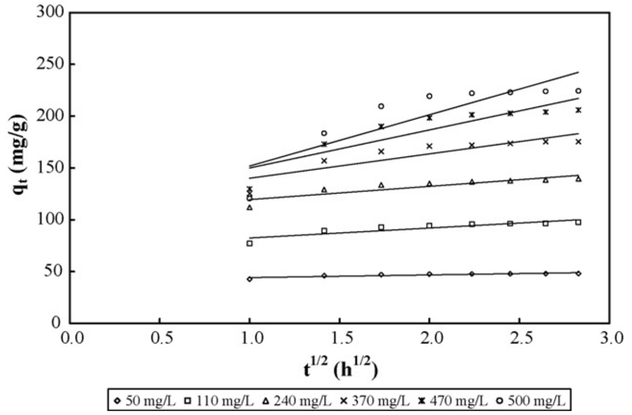


Fig. 8. Intraparticle diffusion plot for adsorption of BB3 onto prepared activated carbon at 30 °C.

[38–41]:

$$q_t = k_{\mathrm{id}} t^{1/2} + C \qquad (13)$$

where q_t is the amount of dye adsorbed at time t, C the intercept and k_{id} is the intraparticle diffusion rate constant. If the Weber–Morris plot of q_t versus $t^{1/2}$ gives a straight line, then the sorption process is controlled by intraparticle diffusion only. However, if the data exhibit multi-linear plots, then two or more steps influence the sorption process. The values of k_{id} and C can be determined from the slope and intercept of the plots q_t versus $t^{1/2}$ (Fig. 8). The k_{id} values were calculated and shown in Table 4. Values of intercept C (Table 4) give an idea about the thickness of the boundary layer, the larger intercept the greater is the boundary layer effect [42].

In order to quantitatively compare the applicability of each model in fitting to data, a normalized standard deviation, Δq, was calculated:

$$\Delta q\,(\%) = 100 \times \sqrt{\frac{\sum [(q_{t,\mathrm{exp}} - q_{t,\mathrm{cal}})/q_{t,\mathrm{exp}}]^2}{n-1}} \qquad (14)$$

Table 4
Intraparticle diffusion rate constants for adsorption of BB3 onto prepared activated carbon at different initial concentration at 30 °C

C_0 (mg/L)	k_{id} (mg/(g h$^{1/2}$))	C (mg/g)	R^2	$q_{\mathrm{e,cal.}}$ (mg/g)	Δq (%)
50	2.58	41.62	0.78	48.91	1.90
110	9.58	72.86	0.80	99.96	3.73
240	12.79	106.66	0.79	142.83	3.50
370	23.60	116.39	0.76	183.13	5.76
470	36.72	113.28	0.79	217.13	7.62
500	49.49	102.42	0.74	242.40	11.89

where the $q_{t,\mathrm{exp}}$ and $q_{t,\mathrm{cal.}}$ refer to the experimental and calculated values and n is the number of data points. Tables 3 and 4 indicate that the pseudo-second-order rate equation agrees well with experimental data, the linear regression coefficient of 1.00 and standard deviation % are less when compared to the pseudo-first-order and intraparticle diffusion models.

3.5. *Performance of prepared activated carbon*

Table 5 shows the comparison of basic dye adsorption of this work and other relevant studies. The most important parameter to compare is the Langmuir $q_{\max}$ value since it is a measure of adsorption capacity of the adsorbent. The value of $q_{\max}$ in this study is larger than those in most of previous works. This suggests that BB3 could be easily adsorbed by rubber seed coat activated carbon used in this work. In addition, the results had shown that the prepared activated carbon could be considered as a promising material to remove basic dye even when compared with commercial activated carbon (Chemviron GW) [10] and commercial granular activated carbon [31] (Table 5). Furthermore, rubber seed coat could be without problems obtained from nature. All these indicate that rubber seed coat activated carbon is a type of cheap and effective adsorbent for BB3.

Table 5
Comparison of basic dye adsorption of this work and previous studies

Dye	Adsorbent	$q_{\max}$ (mg/g)	Reference
Basic blue 3	Rubber seed coat-based activated carbon	227.27	This work
Basic red 46	Chemviron GW[a]	106.00	[10]
Basic yellow	Granular activated carbon produced from coffee grounds	10.00	[31]
Basic yellow	Commercial activated carbon	4.00	[31]
Basic red 46	Sewage sludge-based activated carbon	188.00	[10]
Methylene blue	Hazelnut shell-based activated carbon	8.82	[43]
Methylene blue	Almond shell-activated carbon	1.33	[43]
Methylene blue	Walnut shell-activated carbon	3.53	[43]
Methylene blue	Apricot stones-activated carbon	4.11	[43]
Crystal violet	Phosphoric acid-activated carbon (PAAC) prepared from male flowers coconut tree	60.42	[44]
Crystal violet	Sulphuric acid-activated carbons (SAAC) prepared from male flowers coconut tree	85.84	[44]
Methylene blue	Coir pith carbon	5.87	[45]
Methylene blue	Bamboo dust carbon	143.20	[46]
Methylene blue	Coconut shell	277.90	[46]
Methylene blue	Groundnut shell carbon	164.90	[46]
Methylene blue	Sulphuric acid treated Parthenium (an agricultural waste) carbon (SWC)	39.68	[47]
Methylene blue	Phosphoric acid treated Parthenium (An agricultural waste) carbon (PWC)	88.49	[47]

[a] Powdered commercial activated carbon (Chemviron Carbon, UK).

4. Conclusions

Activated carbon prepared from rubber seed coat, an agricultural solid waste, can be effectively used as adsorbent for the removal of basic dye from aqueous solutions. Langmuir, Freundlich and Temkin isotherm models were used to interpret the adsorption phenomenon of the adsorbate. The equilibrium adsorption data for BB3 was best represented by the Freundlich isotherm. The R_L value showed that activated carbon was favorable for the adsorption of BB3. The suitability of the kinetic models for the adsorption of BB3 on the activated carbon was also discussed. It was found that the adsorption kinetics of dye obeyed pseudo-second-order adsorption kinetics.

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