

Dye removal from wastewater using activated carbon developed from sawdust: adsorption equilibrium and kinetics

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Abstract

Mahogany sawdust was used to develop an effective carbon adsorbent. This adsorbent was employed for the removal of direct dyes from spent textile dyeing wastewater. The experimental data were analysed by the Langmuir and Freundlich models of adsorption. Equilibrium data fitted well with the Langmuir model. The rates of adsorption were found to conform to the pseudo-second-order kinetics with good correlation. The equilibrium adsorption capacity of the sawdust carbon was determined with the Langmuir equation as well as the pseudo-second-order rate equation and found to be >300 mg dye per gram of the adsorbent. The most ideal pH for adsorption of direct dyes onto sawdust carbon was found to be 3 and below. The results indicate that the Mahogany sawdust carbon could be employed as a low cost alternative to commercial activated carbon in the removal of dyes from wastewater.

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1. Introduction

Adsorption is relevant in environmental pollution and protection with reference to water and wastewater treatment [1]. Toxic materials, hazardous ions and dyes from industrial effluents by way of adsorption are of great significance in connection with environmental and human health safety. Adsorption by solids decreases the toxicity of the wastewater or removes non-safe organic materials from industrial effluents, etc. [2,3].

Affluent from the dyeing industry contain highly coloured species; such highly coloured wastes are not only aesthetically displeasing but also hinder light penetration and may in consequence disturb biological processes in water-bodies. In addition, dyes are toxic to some organisms and hence harmful to aquatic animals. Furthermore, the expanded uses of azo dyes have shown that some of them and their reaction products such as aromatic amines are highly carcinogenic [4,5]. Therefore, removal of dyes before disposal of the wastewater is necessary.

Among various treatment technologies, adsorption onto activated carbon has proven to be one of the most effective and reliable physicochemical treatment methodology [6–11]. However, commercially available activated carbons are very expensive. Therefore, there is a need to produce low cost and effective carbons that can be applied to water pollution control. There is a growing interest in using low cost, commercially available materials for the treatment of wastewater. A wide variety of low cost materials, such as clay minerals [12], bagasse pith [13], wood [14], maize cob [15] and peat [16] are being evaluated as viable substitutes for activated carbon to remove dyes from coloured effluents. However, as the adsorption capacities of the above adsorbents are not large, new adsorbents are still under development. Commercial activated carbons are sophisticated in the sense that they are designed for a variety of applications. If low cost non-conventional sources are used to prepare activated carbons for a specific purpose, then they should be economical for wastewater treatment.

The present investigation reports the results of removal of direct dyes from spent textile dyeing wastewater by adsorption onto activated carbon prepared from low cost mahogany sawdust. The objective of the present work is to examine the effectiveness of the prepared activated carbon in remov-

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Table 1
Characteristics of the wastewater samples and the sawdust carbon

Sawdust carbon		Wastewater sample		
Parameter	Value	Parameter	Sample 1	Sample 2
Bulk density (g/ml)	0.38	pH	8.21	8.02
Solid density (g/ml)	3.6	λ_{max} (nm)	570	620
Moisture content (%)	0.52	TSS (mg/l)	198	271
Ash content (%)	6.5	COD (mg/l)	1477.6	1880.6
Particle size (mesh)	200–60	TOC (mg/l)	559.7	734.6
BET surface area (m ² /g)	516.3			
Surface acidity (meq.v/g)	0.02			
Surface basicity (meq.v/g)	2.34			

ing direct dyes from wastewater. The kinetic data and equilibrium data of adsorption studies were processed to understand the adsorption mechanism of the dye molecules onto the sawdust carbon.

2. Experimental

2.1. Materials

A dyeing wastewater collected from a textile dyeing factory in Calcutta was used to evaluate the effectiveness of the prepared sawdust carbon in the treatment of wastewater. Two different coloured wastewater samples, Samples 1 and 2, were used in this study. They were collected directly from the production line. Sample 1 contained Direct Blue 2B dye along with nonionic surfactant (used as wetting agent of cotton cloth) and inorganic salts. Sample 2 contained Direct Green B dye and other chemicals same as that of Sample 1. Detail characteristics of the samples are given in Table 1. The chemical oxygen demand (COD) values of the samples were determined by standard dichromate method [17]. The total organic carbon (TOC) content was determined by Shimadzu TOC-5000A analyser. Because of the presence of suspended solids, the wastewater samples were filtered through glass microfibre filters (Whatman GF/C) prior to experiments.

The adsorbent was prepared from Mahogany sawdust, which was collected from a local sawmill. The material was sieved to obtain the desired size fractions, washed thoroughly with hot distilled water, and then carbonised in a muffle furnace at 500 °C for 1 h. Activation was applied by means of 1.5 kg/cm² steam pressure at 800 °C for 1 h. The activated material thus prepared was then ground and dried in an air oven at 100 ± 5 °C for overnight. Different properties of the adsorbent were determined by standard methods. Some physicochemical characteristics of the adsorbent are summarised in Table 1. The surface area of the activated carbon was determined by the BET (Micromeritics ASAP-2010 adsorption analyser) method. Both surface acidity and surface basicity of the sawdust carbon were determined using acid–base titration methods [18]. Before utilisation, the carbon was thoroughly washed with distilled water several times and then dried in hot air oven at 100 ± 5 °C.

Pure Direct Blue 2B and Direct Green B dyes were purchased from J.K. Chemi-Dyes Enterprises (India) for the preparation of calibration curve, and were used as received. Fig. 1 displays the structure of these two dyes. Wherever required, analytical grade (AR) chemicals were used in the study. Doubly distilled water was used for solution preparation. A Shimadzu (Japan) UV–vis spectrophotometer (160 A) was employed for absorbance measurements using silica cells of path length 1 cm.

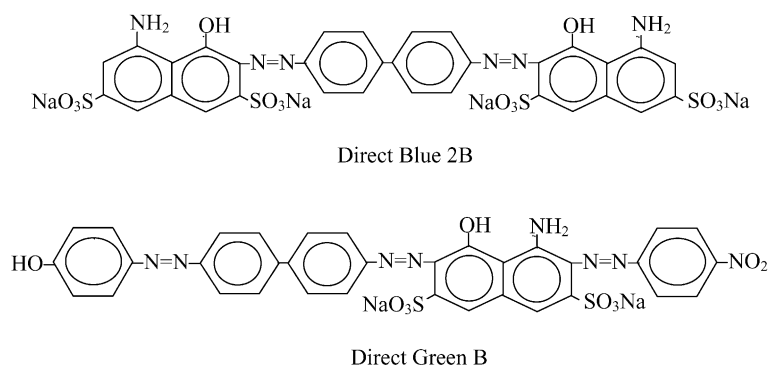


Fig. 1. Structure of Direct Blue 2B and Direct Green B.

2.2. Methods

Batch studies were carried out at the temperature of 30 °C. In the adsorption experiment, weighed quantity of activated carbon was taken in a standard-joint pyrex glass stoppered bottle (250 ml) containing 100 ml wastewater sample. The pH of the wastewater–carbon system was adjusted to the desired value and the mixture was shaken for a predetermined period using a thermostated horizontal shaker operated at 150 rpm. Kinetics of adsorption was determined by analyzing adsorptive uptake of the dye colour at different time intervals. Independent bottles containing 100 ml wastewater sample and 0.06 g activated carbon were used during the kinetic studies to get accurate results for each point on the graph. Isothermal studies were conducted with different doses of adsorbent (0.01–0.3 g) and 100 ml sample by shaking the reaction mixture for equilibrium time. Influence of pH on the adsorption was studied by adjusting the pH of the wastewater–carbon system to different values (2.0–9.0) and analysing the residual colour after equilibrium contact time. Dilute H₂SO₄ and NaOH were used for pH adjustment. The residual dye colour in the reaction mixture was analyzed by centrifuging the reaction mixture and then measuring the absorbance of the supernatant at the wavelength that correspond to the maximum absorbance of the sample. Dye concentration in the reaction mixture was calculated from the calibration curve. The λ_{\max} values of the wastewater samples varied ± 10 nm from the λ_{\max} values of pure dyes at a fixed pH. The amount of dye adsorbed onto the carbons, q_e (mg/g), was calculated by the following mass balance relationship:

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

where C_0 and C_e are the initial and equilibrium liquid-phase concentrations of dye, respectively (mg/l), V the volume of the solution (l), and W is the weight of the carbon used (g).

3. Results and discussion

3.1. Influence of system pH on adsorption capacity

The pH of the system exerts profound influence on the adsorptive uptake of adsorbate molecule presumably due to its influence on the surface properties of the adsorbent and ionization/dissociation of the adsorbate molecule. The variations in removal of dye from wastewater at various system pH are shown in Fig. 2. From the figure, it is evident that the maximum removal of dye colour for both the samples is observed at pH 3 and below. Similar trend of pH effect was observed for the adsorption of Congo Red (Direct Red 28) and Acid Violet on activated carbon prepared from coir pith [19,20], and for the adsorption of Acid Blue 9 onto the mixture of activated carbon and activated clay [21]. The reason could be that the carbon surfaces acquire a basic character on

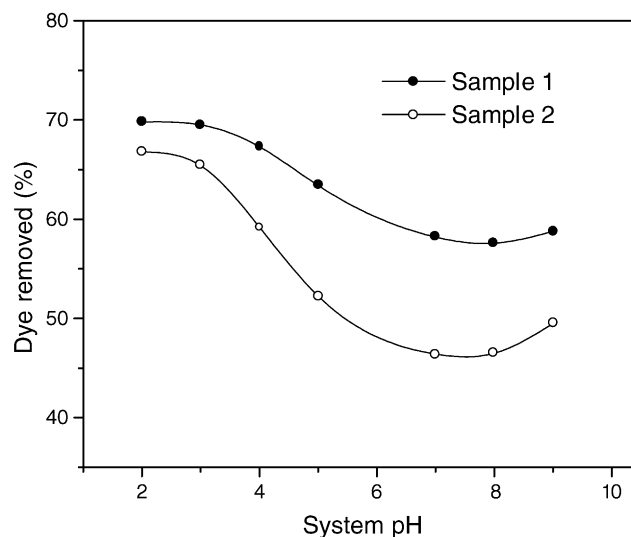


Fig. 2. Effect of system pH on removal of direct dyes by sawdust carbon. Experimental conditions: carbon = 0.6 g/l; contact time = 150 min and temperature = 30 °C.

high temperature (>700 °C) heat treatment [18]. Galiatsatou et al. [22] found that steam activation at 800 °C increases the basic surface groups of activated carbon with increasing activation time. Mattson and Mark [23] reported that the activated carbons produced by high temperature (>750 °C) steam activation are hydrophobic in nature and take on a positive charge by absorbing hydrogen (H⁺) ions when immersed in water. Low pH leads to an increase in H⁺ ion concentration in the system and the surface of the activated carbon acquires positive charge by absorbing H⁺ ions. As the carbon surface is positively charged at low pH, a significantly strong electrostatic attraction appears between the positively charged carbon surface and anionic dye molecule leading to maximum adsorption of dye. As the pH of the system increases, the number of negatively charged sites increases and the number of positively charged sites decreases. A negatively charged surface site on the sawdust carbon does not favour the adsorption of anionic dye molecules due to the electrostatic repulsion. Furthermore, lower adsorption of the direct dyes in alkaline medium is also due the competition from excess OH⁻ ions with the anionic dye molecule for the adsorption sites. In alkaline medium, the extent of dye colour removal is increased as the pH is increased from 8 to 9. This is consistent with the view that at pH ≥ 9 , formations of small coagulated mass of dye colour have been observed for both wastewater samples. This contributes to the extent of dye colour removal at pH 9. It has also been seen that the dye colour of wastewater is precipitated when the pH of the sample solution is raised above 10.

3.2. Adsorption kinetic considerations

The influence of contact time on dye colour removal by activated carbon is presented in Fig. 3. It is evident that

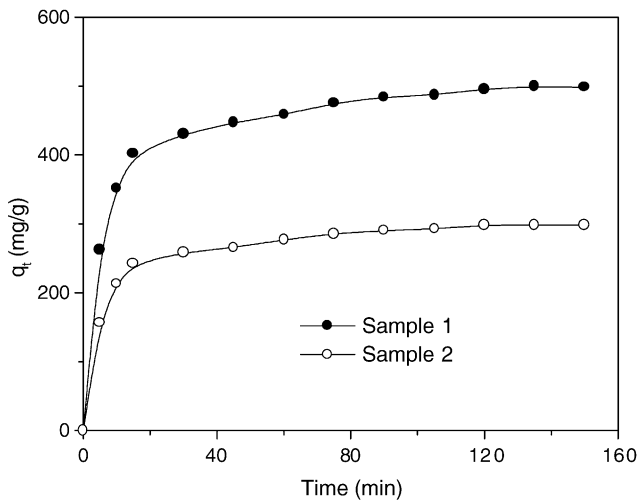


Fig. 3. Time profiles of solid-phase concentrations of dyes for sawdust carbon – dye system. Experimental conditions: carbon = 0.6 g/l; pH 3 and temperature = 30 °C.

the activated carbon prepared from mahogany sawdust is efficient to adsorb direct dyes from wastewater, the process attaining equilibrium gradually. This is due to the fact that activated carbon is composed of porous structure with large internal surface area [7,24,25]. Three consecutive mass transport steps are associated with the adsorption of solute from solution by porous adsorbent [26]. First, the adsorbate migrates through the solution to the exterior surface of the adsorbent particles by molecular diffusion, 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. The time profile of dye uptake is a single, smooth and continuous curve leading to saturation, suggesting also the possible monolayer coverage of dyes on the surface of the sawdust carbon. Fig. 3 shows that the contact time required in attaining equilibrium is almost 120 min for both the samples. A similar phenomenon was observed for the adsorption of Direct Brown 1:1 from aqueous solution on commercial activated carbon and the equilibrium time was 400 min [27].

The kinetic adsorption data was processed to understand the dynamics of adsorption process in terms of the order of rate constant. Kinetic data were treated with the pseudo-first-order kinetic model [28]. The differential equation is the following:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (2)$$

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 (min^{-1}). Integrating Eq. (2) for the boundary conditions $t = 0$ to t and $q_t = 0$ to q_t , gives:

$$\log \frac{q_e}{q_e - q_t} = \frac{k_1}{2.303} t \quad (3)$$

which is the integrated rate law for a pseudo-first-order reaction. Eq. (3) can be rearranged to obtain a linear form:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (4)$$

Values of the rate constant, k_1 , equilibrium adsorption capacity, q_e , and the correlation coefficient, r_1^2 , were calculated from the plots of $\log(q_e - q_t)$ versus t (inset of Fig. 4) for both wastewater samples. Although the correlation coefficients for both samples are found to be higher than 0.98,

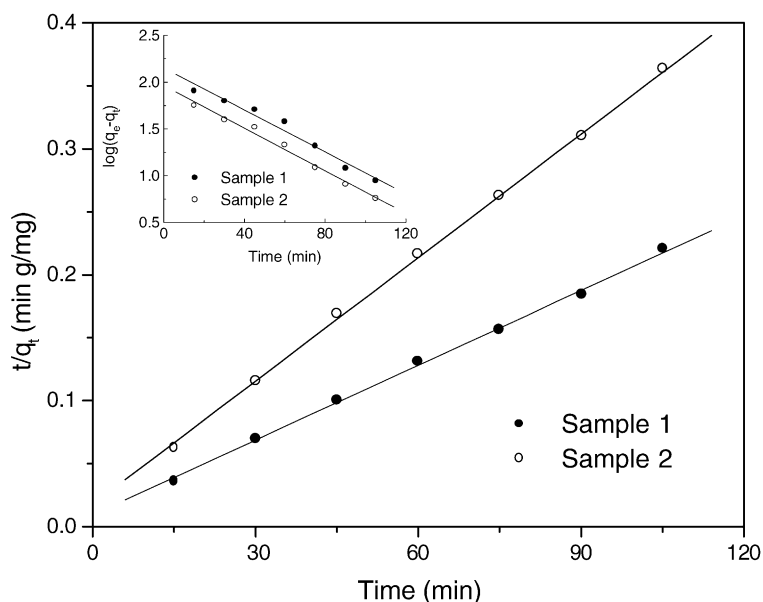


Fig. 4. Pseudo-second-order kinetics for adsorption of direct dyes on sawdust carbon. Inset, pseudo-first-order kinetics for adsorption of direct dyes on sawdust carbon.

Table 2

Pseudo-first- and pseudo-second-order adsorption rate constants and the calculated and experimental q_e values for adsorption of direct dyes on sawdust carbon

Sample	First-order kinetic model				Second-order kinetic model		
	q_e (exp) (mg/g)	k_1 (min^{-1})	q_e (cal) (mg/g)	r_1^2	k_2 (g/mg min)	q_e (cal) (mg/g)	r^2
1	496	0.025	141.6	0.985	0.11	500	0.999
2	298.2	0.026	91.8	0.993	0.09	303	0.999

the calculated equilibrium adsorption capacities do not agree with experimental values (Table 2). This indicates that adsorption of direct dyes onto sawdust carbon is not an ideal pseudo-first-order reaction.

Kinetic data were further treated with the pseudo-second-order kinetic model [29,30]. The differential equation is the following:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (5)$$

where k_2 is the equilibrium rate constant of pseudo-second-order adsorption ($\text{g mg}^{-1} \text{min}^{-1}$). Integrating Eq. (5) for the boundary condition $t = 0$ to t and $q_t = 0$ to q_t , gives:

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + k_2 t \quad (6)$$

which is the integrated rate law for a pseudo-second-order reaction. Eq. (6) can be rearranged to obtain a linear form:

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

If pseudo-second-order kinetics is applicable, the plot of t/q_t versus t should show a linear relationship. The linear plots of t/q_t versus t show a good agreement of experimental data with the pseudo-second-order kinetic model for both

wastewater samples (Fig. 4). The correlation coefficients (r^2) for the second-order kinetic model are higher than 0.99. The second order rate constant, k_2 , and the equilibrium adsorption capacity, q_e , were calculated from the intercept and slope of the plots of t/q_t versus t . The calculated q_e values agree very well with the experimental data (Table 2). These indicate that the adsorption of direct dyes from wastewater on sawdust carbon obeys pseudo-second-order kinetic model. Namasivayam and Kavitha [19] reported similar findings for the adsorption of Congo Red on activated carbon prepared from agricultural waste. Similar phenomenon was observed for the adsorption of Acid Blue 9 onto mixture of activated carbon and activated clay [21], and also in biosorption of reactive dyes on biomass [31,32].

3.3. Adsorption isotherm studies

The adsorption isotherms of various classes of compounds (q_e versus C_e) in different surfaces have been classified according to their shapes. In the literature, four types of isotherm have been reported [33], i.e., (i) the Langmuir type (L) with an initial concavity to the concentration axis, (ii) the S-type with an initial convexity to the concentration axis, (iii) the H-type resulting from extremely strong adsorption and having an intercept on the ordinate and (iv) the C-type

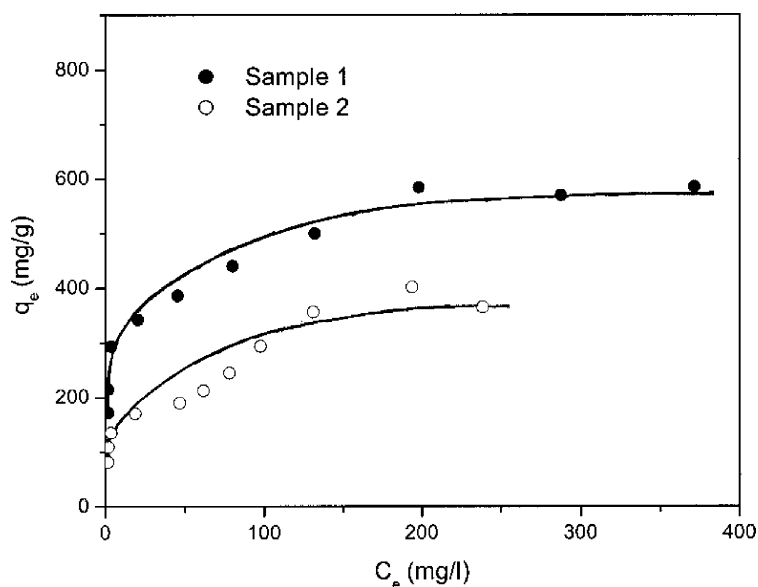


Fig. 5. Isotherm for adsorption of direct dyes on activated carbon prepared from sawdust. Experimental conditions: contact time = 150 min; pH 3 and temperature = 30 °C.

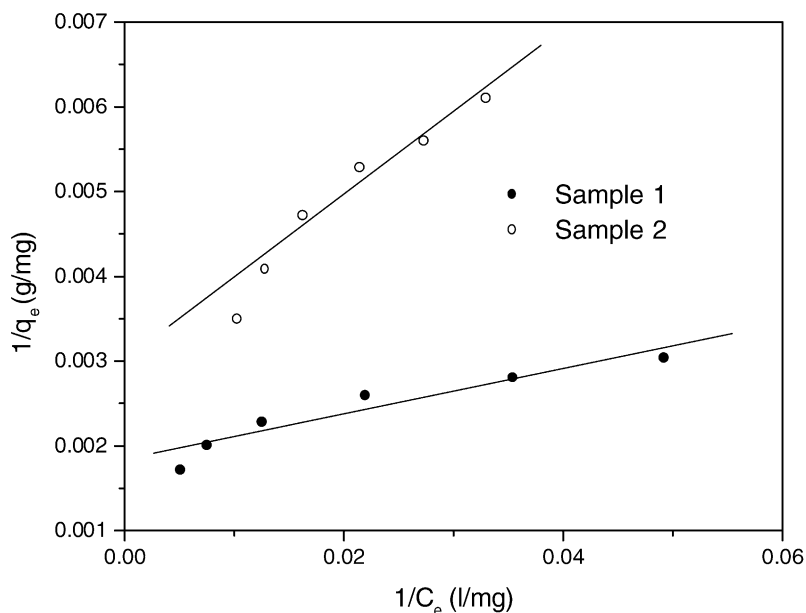


Fig. 6. Langmuir plots for adsorption of direct dyes on sawdust carbon.

having an initial linear portion. Adsorption isotherms expressing the adsorbed amounts as a function of equilibrium concentration for both wastewater samples are presented in Fig. 5. It is evident that the curves are all likely to be a typical Langmuir-type adsorption isotherm.

Two isotherm equations are tested in this work. One is the Langmuir isotherm equation, which has been widely applied to describe experimental adsorption data based on the assumption that maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on adsorbate surface with a constant energy and there is no transmigration of adsorbate in the plane of adsorbate surface. The theoretical Langmuir isotherm equation can be represented as [34]:

$$q_e = \frac{q_{\text{mon}} K_L C_e}{1 + K_L C_e} \quad (8)$$

where K_L is the Langmuir constant related to the energy of adsorption (l/mg) and q_{mon} is the maximum amount of adsorption corresponding to complete monolayer coverage on the surface (mg/g). The constants K_L and q_{mon} can be determined from the following linearised form of Eq. (8):

$$\frac{1}{q_e} = \frac{1}{q_{\text{mon}}} + \frac{1}{K_L q_{\text{mon}}} \frac{1}{C_e} \quad (9)$$

The linear plots of $1/q_e$ versus $1/C_e$ for the wastewater samples show that adsorption follows Langmuir isotherm model (Fig. 6). Confirmation of the experimental

data into Langmuir isotherm model indicates the homogeneous nature of sawdust carbon surface, i.e., each dye molecule/sawdust carbon adsorption has equal adsorption activation energy; the results also demonstrate the formation of monolayer coverage of dye molecule at the outer surface of sawdust carbon. Similar observation was reported for the adsorption of dyes onto activated carbon [19,25,27]. Values of q_{mon} and K_L were calculated from the intercept and slope of the linear plots, respectively, and are presented in Table 3. The equilibrium adsorption capacities evaluated from the Langmuir equation and the pseudo-second-order rate model show that the evaluated values are reasonable. Mohan et al. [27] reported that the q_{mon} value for adsorption of Direct Brown 1:1 from aqueous solution on commercial activated carbon is 7.69 mg/g. A q_{mon} value of 6.72 mg/g was observed by Namasivayam and Kavitha [19] for adsorption of Congo Red on activated carbon prepared from coir pith. Nassar and Magdy [35] found a q_{mon} value of 600, 790, and 648 mg/g for adsorption of BY21, BR22, and BB3, respectively on commercial activated carbon. Juang and Tseng [25] obtained a q_{mon} value of 270.3 and 273 mg/g for adsorption of BR22 and AB25, respectively on activated carbon prepared from bagasse piths by steam activation at 750 °C. Steep slope of the Langmuir plots is indicative of the usefulness of sawdust carbon at

Table 3
Langmuir and Freundlich constants for adsorption of direct dyes on sawdust carbon

Sample	Langmuir isotherm				Freundlich isotherm		
	q_{mon} (mg/g)	K_L (l/mg)	R_L	r^2	K_F	n	r^2
1	518	0.08	0.03	0.980	201.8	5.03	0.954
2	327.9	0.03	0.1	0.982	88.3	3.98	0.944

high concentration and also of the efficiency in column operations.

The essential features of Langmuir adsorption isotherm can be expressed in terms of a dimensionless constant called separation factor or equilibrium parameter (R_L), which is defined by the following relationship [36]:

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

where C_0 is the initial dye concentration (mg/l). The R_L value indicates the shape of the isotherm to be irreversible ($R_L = 0$), favourable ($0 < R_L < 1$), linear ($R_L = 0$) or unfavourable ($R_L > 1$) [37,38]. By processing the above equation, R_L values for investigated dye-adsorbent system are found to be 0.03 for Sample 1 and 0.1 for Sample 2. From the values of R_L , it is confirmed that prepared sawdust carbon is favourable for adsorption of direct dyes from wastewater under the conditions used in this study.

The Freundlich isotherm [39] is the earliest known relationship describing the sorption equation. This fairly satisfactory empirical isotherm can be used for non-ideal sorption that involves heterogeneous surface energy systems and is expressed by the following equation:

$$q_e = K_F C_e^{1/n} \quad (11)$$

where K_F is roughly an indicator of the adsorption capacity and $1/n$ is the adsorption intensity. In general, as the K_F value increases the adsorption capacity of adsorbent for a given adsorbate increases. The magnitude of the exponent, $1/n$, gives an indication of the favorability of adsorption. Values of $n > 1$ represent favourable adsorption condition [40,41]. Eq. (11) may be linearised by taking logarithms:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (12)$$

Linear plots of $\log q_e$ versus $\log C_e$ show that adsorption of direct dyes from wastewater on sawdust carbon also follow the Freundlich isotherm (Fig. 7). Similar observation was reported for adsorption of Congo Red on activated carbon [19], and also for the adsorption of Methyl Orange, Congo Red, Methylene Blue, and Rhodamine B on waste banana peel [42]. Values of K_F and n are calculated from the intercepts and slopes of the plots and are listed in Table 3. The results suggest that direct dyes are favourably adsorbed by activated carbon prepared from sawdust. However, the values of the correlation coefficient (r^2) (see Table 3) indicate that the Langmuir isotherm has been best fitted for the adsorption of direct dyes on sawdust carbon. Moreover, over a wide range of C_e values are necessary to discuss the Freundlich model of adsorption ideally. The values of C_e in this study were from 0.2 to 2.5, too narrow to discuss the Freundlich model of adsorption properly.

It is noteworthy to point out that more than 80% reduction in COD value has occurred for both wastewater samples on treatment with sawdust carbon under the most operating conditions of treatment.

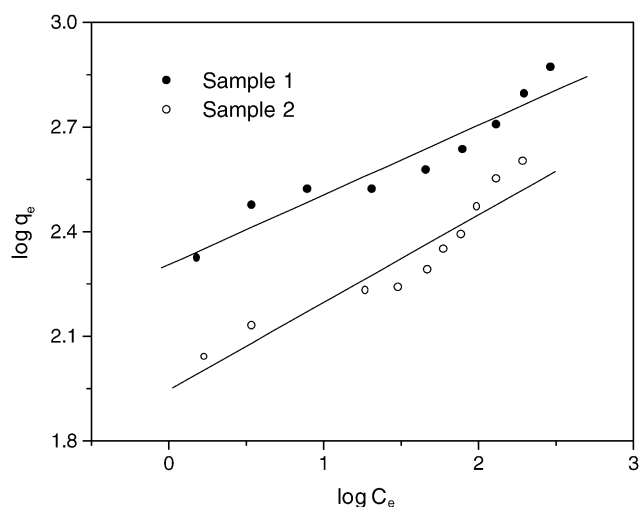


Fig. 7. Freundlich plots corresponding to adsorption of direct dyes on sawdust carbon.

4. Conclusion

The present study shows that activated carbon prepared from low cost material, mahogany sawdust, is considerably efficient for removal of direct dyes from wastewater. The adsorption is highly dependent on contact time, adsorbent dose and pH. The most ideal pH for adsorption of direct dyes on sawdust carbon is 3 and below. The kinetics of direct dye adsorption on sawdust carbon follows the pseudo-second-order model. The equilibrium data fit well in the Langmuir model of adsorption, showing monolayer coverage of dye molecules at the outer surface of sawdust carbon. The value for the maximum adsorption capacity, q_{mon} , is comparable with the values for commercial activated carbon reported in earlier studies. The result would be useful for fabrication and designing of spent textile dyeing wastewater for the removal of dyes. Since the raw material, mahogany sawdust, is freely available the treatment method seems to be economical.

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