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Adsorption of acid dye onto activated carbons prepared from agricultural waste bagasse by $ZnCl_2$ activation

W.T. Tsai [a,*], C.Y. Chang [b], M.C. Lin [a], S.F. Chien [a], H.F. Sun [a], M.F. Hsieh [a]

[a] *Department of Environmental Engineering and Health, Chia Nan University of Pharmacy and Science, Tainan 717, Taiwan, ROC*
[b] *Graduate Institute of Environmental Engineering, National Taiwan University, Taipei 106, Taiwan, ROC*

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Abstract

A series of activated carbons were prepared from agricultural waste sugarcane bagasse by chemical activation with zinc chloride ($ZnCl_2$) as an activating agent at 500°C and 0.5 h soaking time. The Langmuir surface area and total pore volume were used to estimate the average pore diameter of the carbon products. The values of the surface area and pore volume increased linearly with increase in the impregnation ratio (IR) up to 100 wt%. The adsorption capacities of the derived adsorbents for Acid Orange 10 were measured at 20°C and 40°C to gain further insights into the acidic surface oxides of the adsorbent from the results of Fourier transform infrared (FTIR) spectroscopy analysis and pH measurement. Adsorption isotherms of the acid dye on adsorbents prepared were determined and correlated with common isotherm equations. It was found that the Langmuir model appears to fit the isotherm data better than the Freundlich model. The physical properties of these adsorbents were consistent with the parameters obtained from the isotherm equations. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Activated carbon; Sugarcane bagasse; Acid dye; Isotherm

1. Introduction

Synthetic organic dyes present certain hazards and environmental problems. Dye effluents are not only aesthetic pollutants as far as their colors are concerned but may also interfere with light penetration in the receiving bodies of water thereby disturbing the biological process. Further, dye effluents may contain chemicals which exhibit toxic effects toward microbial populations and can be toxic and/or carcinogenic to mammals (Reife, 1993). Therefore, environmental legislation has imposed stringent limits on the concentrations of pollutants, which may be discharged in aqueous effluents from dyestuff manufacturing and textile industries.

In general, dyes are poorly biodegradable or resistant to environmental conditions. The decoloration of dyes containing wastewater can be processed in two ways: chemical and physical methods. Adsorption is an effective method of lowering the concentration of the dissolved dyes in effluents resulting in color removal. Other means of dye removal such as chemical oxidation, coagulation, and reverse osmosis are generally not feasible due to economic considerations (Reife and Freeman, 1996; Nassar, 1997; Slokar and Majcen-Le Marechal, 1998).

Activated carbon is perhaps the most widely used adsorbent for the removal of color (Allen, 1996). Despite the prolific use of this adsorbent throughout the water and wastewater treatment industry, carbon adsorption remains an expensive process due to the high

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[*] Corresponding author. Tel.: +886-6-266-0393; fax: +886-6-266-7323.
E-mail address: wwtsai@mail.chna.edu.tw (W.T. Tsai).

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cost of use of activated carbon (Pollard et al., 1992). Over recent years, this has attracted considerable research into low-cost alternative materials for the production of activated carbon from agricultural wastes for dye removal, such as palm-tree cobs (Khalil and Girgis, 1998), bamboo (Wu et al., 1999a), and plum kernels (Wu et al., 1999b). The feasibility of adsorption on activated carbon for the removal of dissolved organic pollutants has been demonstrated by the adsorption isotherms (Noll et al., 1992; Reife and Freeman, 1996). Also, it is well known that the adsorption capacity depend mainly on the surface area and pore size.

In our previous studies (Tsai et al., 1997, 1998), agrowaste corncob has been found to be a suitable precursor for manufacturing activated carbon by zinc chloride ($ZnCl_2$) activation. The results have shown that an activation temperature of 500°C is the optimal condition for producing activated carbons with large surface area at a pre-pyrolytic heating rate of 10°C min^{-1} and with high impregnation ratios (IRs) under an inert atmosphere of flowing nitrogen. In Taiwan, sugarcane bagasse is the primary agro-industrial waste or by-product. The annual generation of the waste has exceeded 1,000,000 metric tons. Despite many investigations on bagasse or bagasse pitch as an effective adsorbent for the adsorption of organic pollutants (McKay et al., 1987, 1997), the current approaches for the treatment/disposal of this biomass waste are combustion-heat recovery and farmland applications. However, these methods would generate serious environmental problems, such as air pollution and greenhouse gas (e.g., CO_2) emission. A possible utilization of this available biomass resource is to turn it into carbon adsorbent products. This application not only is useful for the removal of environmental organic pollutants (e.g., dyes), but also for the reduction of greenhouse gas CO_2 emission. With respect to the production of activated carbon from agricultural waste sugarcane bagasse, the information published is limited. The objectives of this work were to (1) determine the physical properties (i.e., Langmuir surface area and average pore diameter) of activated carbons prepared from sugarcane bagasse, (2) establish the adsorption isotherms of the resulting carbons in adsorbing a water-soluble acid dye, and (3) study the relationship between the carbons' properties and their ability to adsorb acid dye from aqueous solutions.

2. Materials and methods

2.1. Preparation of raw materials

Sugarcane bagasse was first cracked and crushed to free the pitch and light chaff in a hammer mill, and then sieved to particle size (0.446 mm) with mesh Nos. 30–50. Several IRs (i.e., 25–100 wt%), defined as the ratios of the mass of $ZnCl_2$ to that of bagasse, were applied to prepare the carbon products (denoted as CB).

2.2. Preparation of activated carbon

The apparatus and experimental methods employed in the present work are similar to those for chemical activation with $ZnCl_2$ as reported previously (Tsai et al., 1997, 1998). Pyrolysis temperature was increased at a ramp of 10°C min^{-1} in a N_2 flow (200 cm^3 min^{-1}) to the specified temperature (i.e., 500°C) and then held 0.5 h (i.e., soaking time), which were shown to be the optimal conditions for producing large-surface-area carbons from corncob.

2.3. Characterization measurements of activated carbon

2.3.1. Functional groups analysis

The Fourier transform infrared (FTIR) spectrometry was applied in the characterization of the surface functional groups of the activated carbon prepared. To obtain the observable adsorption spectra, the activated carbon was ground to a very fine powder and then dried at 120°C for at least 24 h. The dilution and homogenization of the fine carbon sample to approximately 0.1 wt% with KBr (spectroscopic grade) were carried out with additional grinding. Discs (12.7 mm I.D. and approximately 1 mm thickness) were prepared in a manual hydraulic press (15 tons max.) at 10 tons for a pressing time of 0.5 min. The spectra were measured and recorded (2500–500 cm^{-1}) on a Bomen DA8.3 spectrometer with a resolution of 2 cm^{-1}.

2.3.2. Determination of pH

The pH of the carbon may be an important parameter of interactions between the adsorbate containing fluid and the carbon adsorbent, which contains other noncarbonaceous constituents. Therefore, the activated carbon prepared was also characterized by measuring the pH. The pH value of the best carbon product (i.e., CB-100) was determined by using the standard method of ASTM D-3838 (ASTM, 1996a).

2.3.3. Determination of surface area and total pore volume

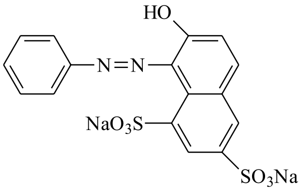
Physical property analyses of the resulting activated carbons were measured at liquid nitrogen temperature (i.e., −196°C) using an ASAP 2010 apparatus (Micromertics) in order to obtain information on the monolayer adsorption capacity of the carbons. The surface areas of the samples were obtained by means of standard methods (i.e., Langmuir). The total pore volume (V_t) was calculated by converting the amount of nitrogen gas adsorbed at a relative pressure ca. 0.975 to the volume of liquid adsorbate. The average pore diameter (D_p) was estimated by assuming a straight, cylindrical, and uninterconnected pore structure (Smith, 1981); that is,

$$D_P = 4V_t/S_L, \tag{1}$$

where S_L is the Langmuir surface area based on monolayer capacity.

2.4. Adsorption of acid dye

The adsorbate used in the experiments was an acid dye, i.e., Acid Orange 10 (C.I. 16230, F.W. 452.38 g mol^{-1}), which was purchased from Aldrich. The estimated molecular size in two dimensions of the dye is 13.8 × 8.3 Å (Tamai et al., 1999). Its formula is



Adsorption capacities of the acid dye were determined by using the standard method of ASTM D-3860 (ASTM, 1996b). In each experiment, 50 mg of fine sample (less than 44 μm, i.e., opening size of mesh No. 325) were used after degassing at 105°C for 24 h in the oven. Adsorption was carried out by shaking (at 110 rpm) a fixed mass (i.e., 50 mg) of dry sample with fixed volumes (i.e., 0.025 dm^3) of acid dye solutions at different initial concentrations in a thermostatted shaker bath at 20°C and 40°C. The acid dye solutions were contacted for 20 h, which was well in excess of the 2 h contact time requirement to reach equilibrium in the standard method as shown previously. The concentrations of aqueous solutions of Acid Orange 10 were varied in the range 5–25 mg dm^{-3}. Concentration analysis of the dye solutions after vacuum filtration with fiber membrane was carried out by a spectrophotometer (Shimadzu UV-1201) at a λ_{max} of 475 nm. All the experiments were carried out in duplicate. The amount of dye adsorbed, q_e (mg g^{-1}), was determined as follows:

$$q_e = (C_0 - C_e)V/W, \tag{2}$$

where C_0 and C_e are the initial and equilibrium liquid-phase concentrations of dye solution (mg dm^{-3}), respectively, V is the volume of dye solution (dm^3), and W is the mass of dry carbon sample used (g).

3. Results and discussion

3.1. Chemical characterization of the activated carbons

3.1.1. Function groups analysis

In the present study, the FTIR spectra of the carbon product CB-100 are shown in Fig. 1. It is seen that the

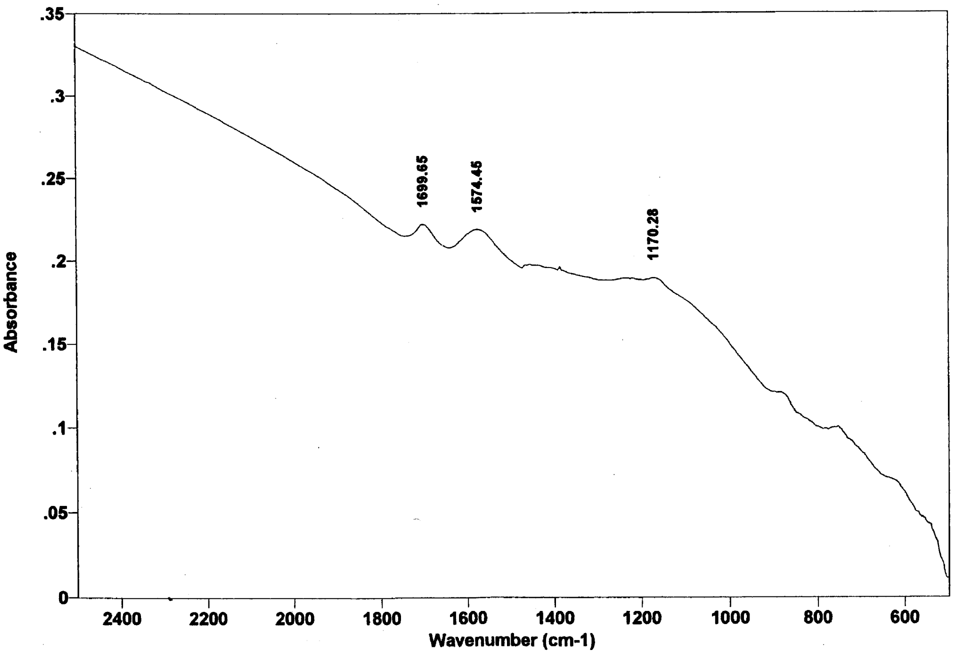


Fig. 1. FTIR diagram of the carbon product CB-100.

absorbance bands were observed to have peaks at 1170.28, 1574.45 and 1699.65 cm^{-1}. Most of these bands have been reported by other investigators for different carbon materials (Ishizaki and Marti, 1981; Kinoshita, 1988). The most significant absorbance peak is at 1574.45 cm^{-1}. This should be the characteristic one of C=O in the quinone structure. The peak of 1699.65 cm^{-1} is the characteristic of the C=O stretching vibration of lactonic and carbonyl groups. The absorbance peak at 1170.28 cm^{-1} may be associated with the stretching vibration of the C—O— bands due to the phenolic structure. The oxygen complexes on the surface may add a polar nature to activated carbons, e.g., hydrophilicity and acidity (Suzuki, 1990).

3.1.2. *pH value of activated carbon*

The pH value of the carbon product CB-100 is about 2.3. The acidity measured should arise from acidic functional groups, e.g., carboxyl and phenolic hydroxyl groups on the surface of activated carbon (Suzuki, 1990). Obviously, this is consistent with the observations in the results of FTIR analysis.

3.2. *Physical properties of the activated carbons*

The data in Table 1 indicate the Langmuir surface area (S_L) and the total pore volume (V_t) of the resulting activated carbons which were prepared from bagasse with IRs of 25–100 wt% $ZnCl_2$ at an activation temperature of 500°C and a soaking time of 0.5 h. The average pore diameters (D_p) of the carbon products are also listed in Table 1. These results reveal the following points.

1. The values of the S_L and the V_t increase proportionally with increase in IR, i.e., CB-100 > CB-75 > CB-50 > CB-25. This means that with increasing IR, more surface area is developed. The activated carbons with higher surface area and/or total pore volume are expected to be the excellent adsorbents for the removal of organic pollutants in aqueous solution.
2. The values of D_p indicate a characteristic of microporous structure (i.e., pore diameter <20 Å) for CB adsorbents (except CB-25). This is consistent with the data of N_2 adsorption isotherms of Langmuir type (Gregg and Sing, 1982). The prepared carbon also exhibits a narrow pore size distribution that is essentially microporous, as illustrated in Fig. 2. Fig. 2(a) illustrates the pore size distribution of activated carbon CB-100 based on the differential pore volume of Barrett–Joyner–Halenda (BJH) adsorption (Gregg and Sing, 1982). Fig. 2(b) shows the micropore size distribution by using the micropore analysis (MP) method (Gregg and Sing, 1982). It is noted that the micropores generally offer a dominant contribution to the adsorption capacity for adsorbate molecules small enough to penetrate. However, transport within these pores can be severely limited by steric effects (Suzuki, 1990).
3. The results show that the large surface area and total pore volume of the best carbon (i.e., CB-100) are about one-half of those of the commercial activated carbons in the liquid-phase adsorption (Table 1). It is noted that total pore volume is usually used as an indication for the adsorption capacity, because its measurement is based on converting the amount of adsorbate (i.e., nitrogen) adsorbed at a relative pressure ca. 0.975 to the volume of liquid nitrogen. As a preliminary evaluation, we demonstrated that the adsorption capacity of adsorbent PCB for acid dye is larger than that of the present resulting carbons in the adsorption experiments.

3.3. *Adsorption isotherms*

In order to optimize the design of an adsorption system to remove the dye, it is important to establish the most appropriate correlations for the equilibrium data for each system. Two isotherm equations have been tested in the present study: Langmuir and Freundlich models. The applicability of the isotherm equations is compared by judging the correlation coefficients, R^2.

Table 1
Description of activated carbons studied and textural characteristics

Activated carbon ID	Langmuir surface area[a] S_L (m^2 g^{-1})	Total pore volume[b] V_t (cm^3 g^{-1})	Average pore diameter[c] D_p (Å)
CB-25[d]	4.86	0.00334	27.5
CB-50	287	0.107	14.9
CB-75	526	0.194	14.8
CB-100	790	0.288	14.6
PCB[e]	1400 (ca.)	0.519	14.2

[a] Calculated from monolayer capacity based on Langmuir model.
[b] Estimated from the pores less than about 80 nm at relative pressure of 0.97.
[c] Computed from the Langmuir surface area (S_L) and total pore volume (V_t) (Smith, 1981), i.e., $D_p = 4V_t/S_L$.
[d] Denotes the carbon prepared from bagasse with impregnation ratio of 25 wt%.
[e] See Tsai and Chang (1994).

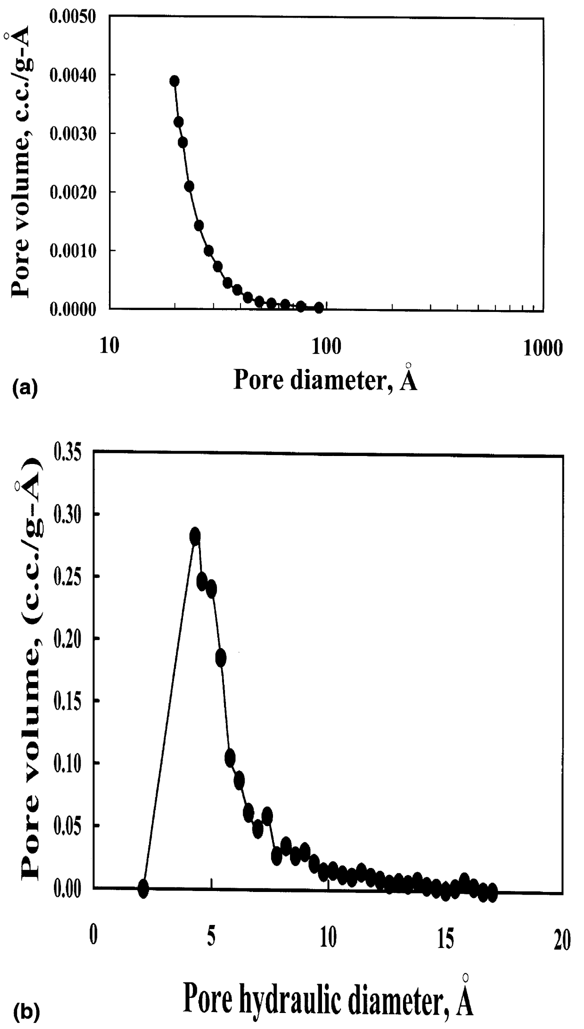


Fig. 2. Differential pore size distribution of the carbon sample CB-100: (a) Barrett–Joyner–Halenda (BJH) adsorption pore (>20 Å) distribution; (b) micropore (<20 Å) distribution from the micropore analysis (MP) method.

3.3.1. Langmuir model

The Langmuir equation is the most widely used two-parameter equation, commonly expressed as

$$C_e/q_e = 1/(K_L q_m) + (1/q_m)C_e. \qquad (3)$$

In Eq. (3), C_e and q_e are as defined in Eq. (2), K_L is a direct measure for the intensity of the adsorption process ($\mathrm{dm^3\ mg^{-1}}$), and q_m is a constant related to the area occupied by a monolayer of adsorbate, reflecting the adsorption capacity ($\mathrm{mg\ g^{-1}}$). From a plot of C_e/q_e vs. C_e, q_m and K_L can be determined from its slope and intercept.

Table 2 presents the results of the Langmuir isotherm fits of Acid Orange 10 to the measured adsorption capacity data for CB adsorbents at 20°C and 40°C. The Langmuir isotherm appears to fit the data reasonably well (Fig. 3), as reflected with correlation coefficients (R^2) in the range of 0.98–1.00. Table 2 indicates the monolayer adsorption capacity, q_m, and Langmuir constant, K_L, increase with increasing surface area as expected. This tendency is reasonable since the adsorption affinity and monolayer adsorption capacity will be enhanced with increasing surface area of the CB adsorbents (Table 1). However, comparing the values of K_L and q_m at 20°C and 40°C, one sees that the adsorption intensity and adsorption capacity at 40°C are higher than those at 20°C. It implies that the enthalpy change (ΔH) had positive values, indicating that the adsorption process of Acid Orange 10 on CB adsorbents was endothermic, which was opposite to the exothermic adsorption usually encountered, and thereby suggesting the possibility of bonding between the adsorbate and the carbon products. Thus, it should be pointed out that sulfonic ($\text{—}SO_3^-$) and/or phenolic ($\text{—}O^-$) groups of Acid Orange 10 may interact with the acidic oxide sites on the surface of the carbon CB-100 which are observed in Fig. 1.

Fig. 4 shows the relationships between q_m (Table 2) and Langmuir surface area (S_L, Table 1). It is evident

Table 2
Parameters in Langmuir adsorption model of Acid Orange 10[a] on CB adsorbents

Temperature (°C)	CB adsorbent	K_L ($\mathrm{dm^3\ mg^{-1}}$)	q_m ($\mathrm{mg\ g^{-1}}$)	Correlation coefficient R^2
20	CB-25	0.46	2.34	0.9892
20	CB-50	0.66	2.84	0.9912
20	CB-75	1.31	4.06	0.9918
20	CB-100	1.32	4.86	0.9912
40	CB-25	0.38	3.16	0.9840
40	CB-50	0.58	3.92	0.9887
40	CB-75	2.23	5.02	0.9990
40	CB-100	3.42	5.78	0.9995

[a] The concentrations are in the range of 5–25 mg $\mathrm{dm^{-3}}$.

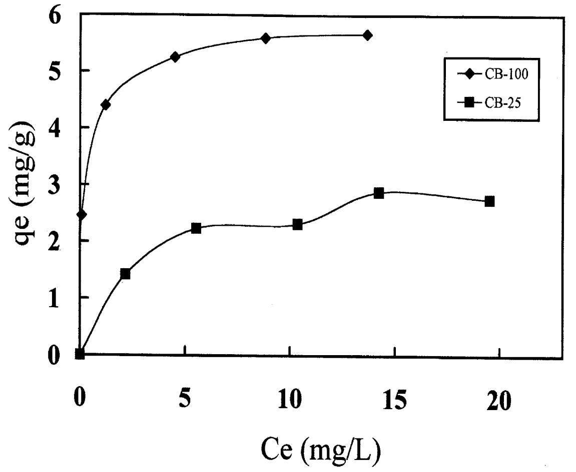


Fig. 3. Adsorption isotherms of the acid dye on the carbon samples CB-25 and CB-100 at 40°C.

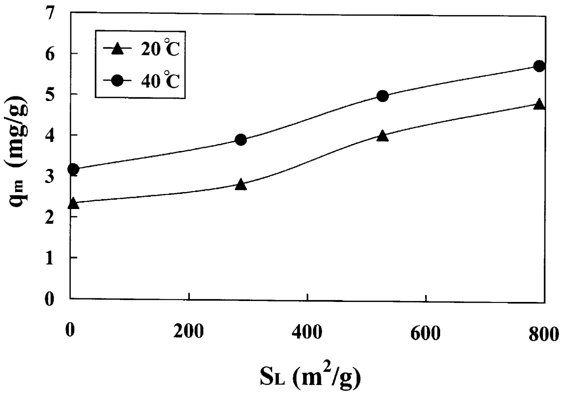


Fig. 4. Dependence of monolayer adsorption capacities (q_m) of the acid dye on Langmuir surface area (S_L) of the carbon products.

that the nearly linear relationships were obtained for the adsorption of Acid Orange 10, because the molecular size (13.8 × 8.3 Å) of the dye is lower than the average pore size (14.6–27.5 Å) of CB adsorbents (Table 1). Therefore, the monolayer adsorption capacities of activated carbons are mainly dependent upon physical properties such as Langmuir surface area.

3.3.2. Freundlich model

The Freundlich isotherm is an empirical equation and shown to be satisfactory for low concentrations. The equation is commonly given by

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

where K_F is a constant for the system, related to the bonding energy. K_F can be defined as the adsorption or distribution coefficient and represents the quantity of dye adsorbed onto carbon adsorbents for a unit equilibrium concentration (i.e., $C_e = 1$ mg dm^{-3}). 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 (Haghseresht and Lu, 1998). A value for $1/n$ below one indicates a normal Langmuir isotherm while $1/n$ above one is indicative of cooperative adsorption (Fytianos et al., 2000). A plot of $\ln q_e$ vs. C_e enables the empirical constants K_F and $1/n$ to be determined from the intercept and slope of the linear regression.

Table 3 presents the effect of temperature and CB adsorbents on the Freundlich parameters, indicating the satisfactorily good correlation between the model predictions and the experimental data. Obviously, it can be seen that the Langmuir model yields a somewhat better fit than the Freundlich model, comparing the results of Table 2 to those of Table 3. It is also seen (Fig. 5) that the values of K_F increase with surface area of CB adsorbents at the same temperature, because the magnitude of K_F indicates a measure of the adsorption capacity. As also illustrated in Table 3, the values of $1/n$ are in the range of 0.12–0.31, which indicates favorable adsorption (Adamson, 1990). However, the values increase with temperature. The Freundlich model provides further insight into the adsorption behaviors of the dye. The slopes of the plots, $1/n$, are observed to decrease in

Table 3
Parameters in Freundlich adsorption model of Acid Orange 10[a] on CB adsorbents

Temperature (°C)	CB adsorbent	K_F [mg g^{-1} (dm^3 mg^{-1})$^{1/n}$]	$1/n$	Correlation coefficient R^2
20	CB-25	1.1851	0.1905	0.9683
20	CB-50	1.6971	0.1440	0.9631
20	CB-75	2.6070	0.1394	0.9885
20	CB-100	3.1982	0.1293	0.9862
40	CB-25	1.1876	0.3062	0.9106
40	CB-50	1.8511	0.2316	0.9957
40	CB-75	3.2914	0.1638	0.9831
40	CB-100	3.9142	0.1685	0.9676

[a] The concentrations are in the ranges of 5–25 mg dm^{-3}.

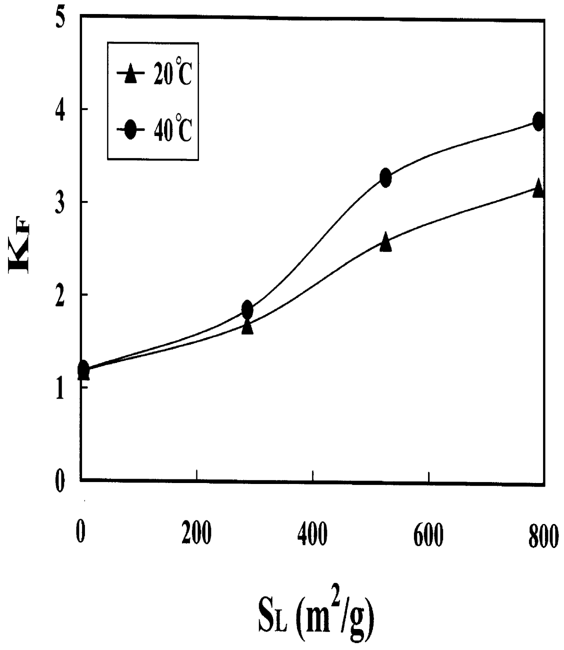


Fig. 5. Dependence of Freundlich constant (K_F) of the acid dye on Langmuir surface area (S_L) of the carbon products.

the order of CB-25 > CB-50 > CB-75 > CB-100, which illustrates that CB-100 is more favorable for adsorption than the others and are in the same trend as K_L, for which CB-100 has the highest value of K_L determined from the Langmuir model. This also indicates that the effect of surface heterogeneity is more pronounced, for CB-100, when a stronger adsorbate–adsorbent bond is formed.

For predicting the favorability of an adsorption system, the Langmuir equation, like the Freundlich equation, can also be expressed in terms of a dimensionless separation factor, R, defined as (McKay et al., 1997)

$$R = 1/(1 + K_L C_0). \tag{5}$$

In Eq. (5), K_L and C_0 are as defined in Eqs. (2) and (3), respectively. For $R = 1$ and 0, the adsorptions are linear and irreversible, respectively. A low value of R favors adsorption. According to the values of K_L and C_0 obtained (Table 2), the present adsorption systems are all favorable, especially for the resulting carbons (e.g., CB-100) with high surface area.

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