Pore-Size Effects on Activated-Carbon Capacities for Volatile Organic Compound Adsorption

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Gas-phase adsorption of acetone and n-hexane by activated carbons with different pore structures was investigated. The carbons were prepared from a bituminous coal with KOH activation. Increasing the activation temperature increased both the porosity and pore size. The equilibrium adsorption capacity for the organic compounds increased with the carbon porosity, but not proportionally. The percentage of pore volume utilized showed a decreasing trend with the porosity development for acetone adsorption, while an increasing trend was observed for n-hexane. By incorporating pore size distribution with the Dubinin-Radushkevich equation using an inverse proportionality between the micropore size and adsorption energy, the isotherms for adsorption onto different carbons can be well predicted. Simulations indicated that the adsorption energy, which is an inverse function of the micropore size, determines the adsorption capacity. Different effects of porosity development were observed for different adsorbates.

Introduction

The increasing awareness of the necessity for environmental safety and pollution control has opened new prospects for the adsorption process. In many industrial chemical plants, such as printing, coating, textile dyeing, and polymer processing, volatile organic compounds (VOCs) are commonly used and emitted. To recover them and/or to reduce their concentrations in effluent streams due to environmental concerns, one of the most effective and versatile methods is adsorption. Activated carbons have been frequently used to remove chemical species from the gas or liquid phase through their excellent adsorption capacities, which are closely related to a large surface area and pore volume. There has been a large number of investigations using activated carbon for gaseous VOC removal (for example, Gregg and Sing, 1982; Bansal et al., 1988; Noll et al., 1992; Admadpour and Do, 1995; Rodríguez-Reinoso, 1997).

Activated carbons are produced from a variety of raw materials, the most common of which are coal, wood, peat, and coconut shells (Wigmans, 1989; Ibarra et al., 1991; Muñoz-Guillena et al., 1992; Laine and Yunes, 1992; Jagtoyen and Derbyshire, 1993; Rodríguez-Reinoso et al., 1995; Teng and Lin, 1998). Depending on the raw materials, activated carbons would have different surface characteristics, including surface functional groups, surface area, porosity, and pore-size distribution. Activated carbons prepared from coals with physical activation using steam are widely available in the market (Wilson, 1981; Rodríguez-Reinoso, 1997). The use of chemically activated carbons for VOC removal has been rarely discussed in the literature. Chemical activation with KOH has been known to be very effective in creating carbon porosity (Evans et al., 1999). In our recent research (Teng and Hsu, 1999), an Australian bituminous coal was chosen as the precursor of activated carbon and high-porosity carbons were produced via a chemical-activation route using KOH as the chemical reagent. Carbons activated with KOH will be used as the adsorbents for VOCs in this study.

It is well known that the extent of activation affects the pore structure of the resulting carbons (Stoeckli and Ballerini, 1991; Merchant and Petrich, 1993; Cal et al., 1994; Mangun et al., 1998; Teng and Lin, 1998; Lee and Reucroft, 1999), but how the efficiency for VOC removal can thus be influenced has not been extensively explored, especially for carbons derived from chemical activation. To shed light on this aspect, two commonly observed organic compounds, acetone and *n*-hexane, which represent compounds with different molecular polarities, are chosen as the adsorbates in the

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present study. Adsorption experiments are conducted using carbon adsorbents from KOH activation to different extents. The equilibrium adsorption data are fitted to well-developed models, and important parameters are obtained for the different carbons to assess the accessibility of the micropores to these VOCs.

Experimental Studies

The activated carbons employed were prepared from an Australian bituminous coal, Black Water (BW, high volatile A in ASTM classification). The preparation consisted of KOH impregnation at a KOH/coal weight ratio of 4.25 followed by carbonization at different temperatures. Details of the preparation process have been described elsewhere (Teng and Hsu, 1999).

The specific surface area and pore volume of these carbon adsorbents were determined by N_2 adsorption. Nitrogen isotherms were measured using an adsorption apparatus (Micromeritics, ASAP2010) at −196°C. The specific surface area was calculated by the standard Brunauer−Emmett−Teller (BET) method and designated as S_{BET}. The Dubinin−Radushkevich (DR) equation was employed to obtain the micropore volume (W_0). The total pore volumes (V_t) were estimated on the basis of the N_2 volume adsorbed at a relative pressure of 0.98. Consequently, the subtraction of the micropore volume (determined from the DR equation) from V_t would provide the volume of the mesopores (Rodríguez-Reinoso et al., 1995).

Both the organic compounds, acetone and *n*-hexane, are GR grade solvents supplied by Riedel-de Haën Chemicals. A standard thermogravimetric analyzer (TGA, Perkin-Elmer TGA7) was used for studying the adsorption of the VOCs onto the carbon adsorbents. Experiments were performed under a VOC/He gas flow of 100 cm^3/min at 1 atm total pressure, with the VOC relative pressure varying between 0 and 0.15. The carbon samples (about 8 mg) were placed in a platinum pan suspended in the heated zone of a quartz tube. A thermocouple placed within a few millimeters of the pan served to show its temperature. Before conducting each experiment, the surface of the samples was cleaned with heat treatment at 350°C in extra-high-purity helium for 1 h. The adsorption experiments were carried out at 30°C. Mass uptake of the organic compounds during adsorption was continuously monitored for a period of at least 2 h, during which time the rate of mass gain was found to be negligible.

Results and Discussion

Surface characteristics of activated carbons

The adsorption isotherms of N_2 on the activated carbons at −196°C are shown in Figure 1. Each carbon has been designated using the nomenclature of the precursor (BW) followed by the carbonization temperature. All the isotherms are of type I according to IUPAC classification (Gregg and Sing, 1982), reflecting the domination of micropores in the pore structure. An increase in adsorption capacity with the carbonization temperature due to enhanced KOH activation can also be observed.

The surface characteristics of the carbons determined from the N_2 isotherms are listed in Table 1. As expected from Figure 1, the BET surface area and the pore volumes are all increasing functions of the carbonization temperature. The mesopore fraction of the pore volume is shown to increase with the temperature due to pore widening upon KOH etching (Teng and Wang, 2000).

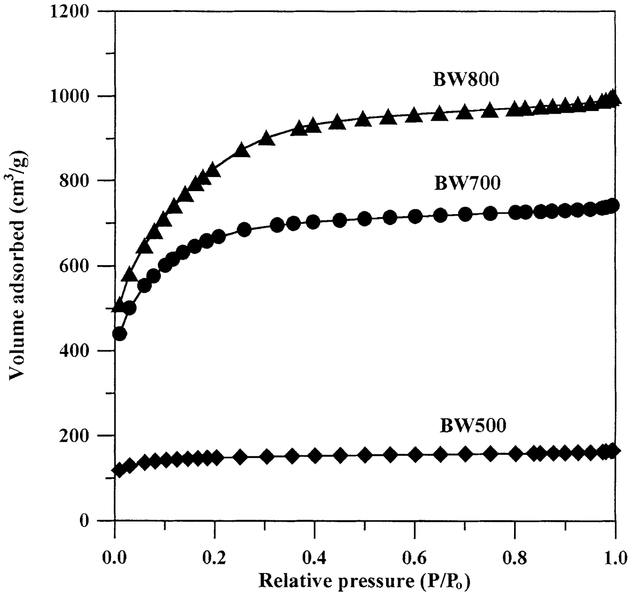

Figure 1. Adsorption isotherms of N_2 on the BW activated carbons with KOH activation at different temperatures.

Because of the domination of micropores in the pore structure, the micropore-size distributions of these carbons were further determined from the application of the Dubinin−Stoeckli (DS) equation to the N_2 adsorption isotherms. The DS equation has been widely employed in the determination of the distribution of micropore sizes in carbons (Dubinin and Stoeckli, 1980; Dubinin, 1989). The DS model assumes that the micropore volume has a normal Gaussian distribution with respect to the size. The model can be presented by

$$\frac{dW}{dX} = \frac{W_0}{\delta\sqrt{2\pi}} \exp\left[-\frac{(X_0 - X)^2}{2\delta^2}\right] \tag{1}$$

where W is the cumulative micropore volume, X the half-width of siltlike micropores, W_0 the micropore volume determined from the DR equation, X_0 the micropore half-width at the distribution curve maximum, and δ the dispersion parameter. The Gaussian distribution of micropore size sug-

Table 1. Surface Characteristics of Activated Carbons Prepared from BW Coal with Different Extents of KOH Activation

Sample	S_{BET} (m^2/g)	V_t (cm^3/g)	W_0 (cm^3/g)	Mesopore Fraction (%)
BW500	504	0.25	0.23	7
BW700	2,340	1.14	0.98	13
BW800	3,010	1.53	1.17	24

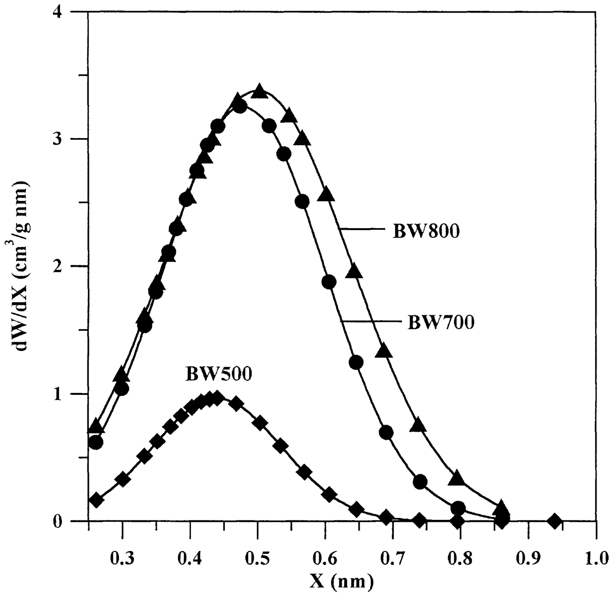

Figure 2. Micropore-size distribution of the activated carbons determined from the Dubinin–Stoeckli equation.

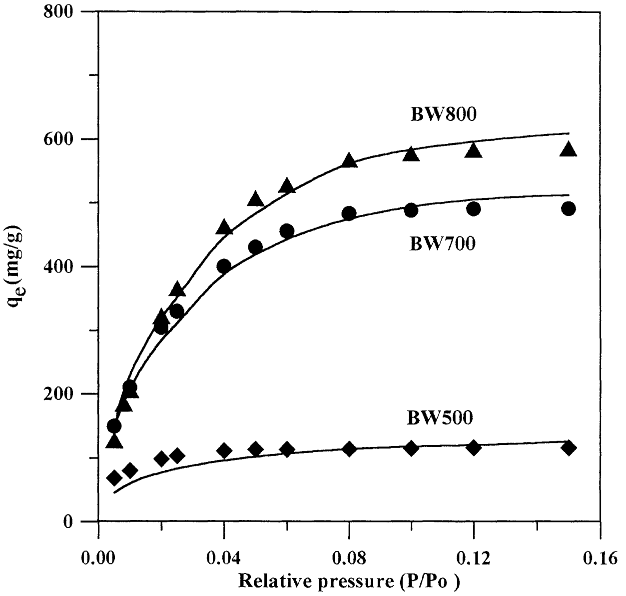

Figure 3. Equilibrium adsorption isotherms of acetone onto different activated carbons at 30°C.

The symbols are experimental data, and the solid line curves are the predictions from Eq. 6.

gested in the DS equation is based on the assumption of random formation of the pores. In a recent study (Daley et al., 1996), the DS equation has shown excellent correlation when compared to direct observation of the porosity using scanning tunneling microscopy (STM). Although the STM results reveal a non-Gaussian distribution about the average pore width, the features of the porosity and the trends with the activation level measured using STM are surprisingly similar to those calculated from the DS equation.

The micropore-size distributions of the carbons were thus determined, using the least-square formulation combined with the numerical method to obtain the optimal values of X_0 and δ in Eq. 1. The results are shown in Figure 2, and the parameters of the distribution curves are summarized in Table 2. As indicated from the data, X_0 increases and the pore-size distribution broadens with increasing carbonization temperature. These distribution curves will be employed to interpret and predict the results for organic compound adsorption in the following discussion.

Organic compound adsorption isotherms

The adsorption isotherms of acetone and *n*-hexane onto the activated carbons at 30°C are shown in Figures 3 and 4, respectively. Typical type I adsorption isotherms were obtained. In the range of concentrations studied, the adsorption capacity at a specified concentration generally increases with the surface area and pore volume, which are increasing functions of the carbonization temperature. Comparing the isotherms in Figures 3 and 4 with the surface characteristics of the activated carbons in Table 1 reveals that, at first glance, the equilibrium adsorption capacity is simply controlled by the area or volume available in the pores, and the pore size

Table 2. Most Suitable Parameters of the Dubinin–Stoeckli Equation for the Activated Carbons

Sample	X_0 (nm)	δ (nm)
BW500	0.44	0.095
BW700	0.47	0.12
BW800	0.50	0.14

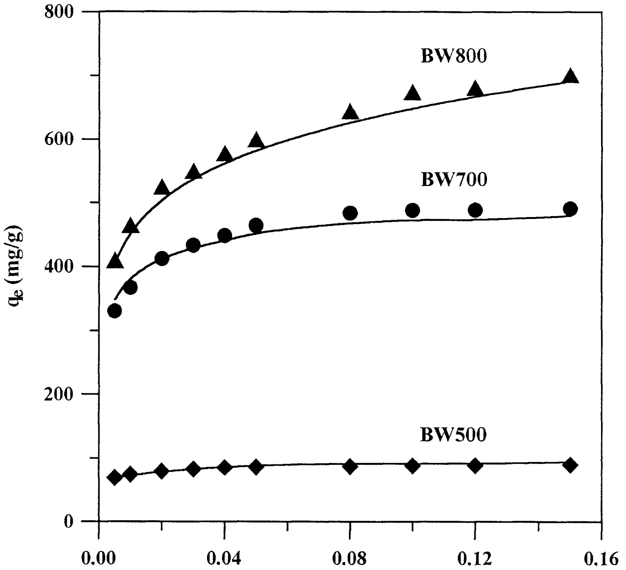

Figure 4. Equilibrium adsorption isotherms of *n*-hexane onto different activated carbons at 30°C.

The symbols are experimental data, and the solid line curves are the predictions from Eq. 6.

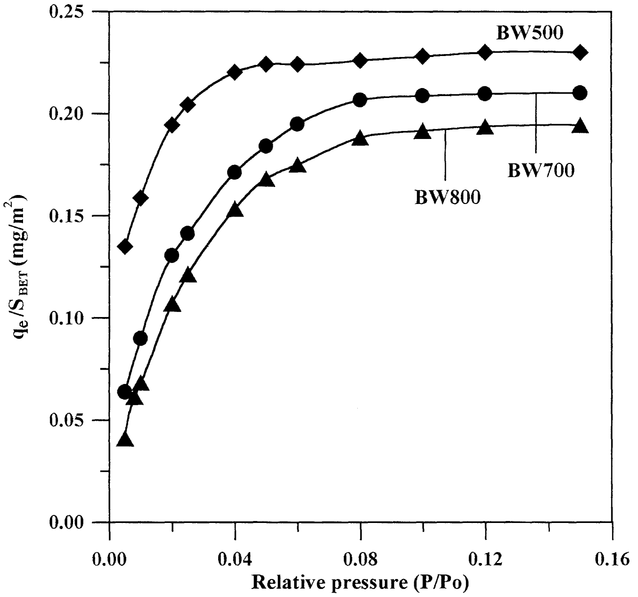

Figure 5. Equilibrium capacities per unit BET area (q_e/S_{BET}) of the carbons for the adsorption of acetone at 30°C.

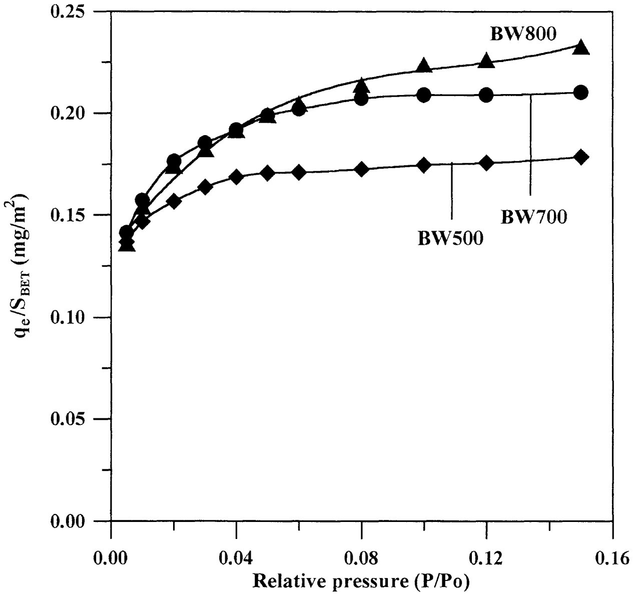

Figure 6. Equilibrium capacities per unit BET area (q_e/S_{BET}) of the carbons for the adsorption of *n*-hexane at 30°C.

shows little influence on the adsorption. A decrease in adsorption capacity with increasing carbon porosity has been reported (Cal et al., 1994; Mangun et al., 1998), and this has been attributed to the lower overlapping potential for adsorption onto higher porosity carbons that contained large-size pores.

The adsorption data given in Figures 3 and 4 were recalculated in terms of capacity per unit BET surface area of the adsorbents, or q_e/S_{BET}. The reason for assessing the adsorption capacity in this manner is because BET surface area and pore volume are strongly related and vary widely for all adsorbents. The BET surface area, hence, for some practical purposes, plays an important role in evaluating the adsorption performance of an adsorbent. On this basis, the adsorption data are displayed in Figures 5 and 6. It is of interest to note that the adsorption performances demonstrated in Figures 5 and 6 are different from those shown in Figures 3 and 4. The value of q_e/S_{BET} for acetone adsorption is a decreasing function of the extent of activation, while that for *n*-hexane shows an increasing trend. The decrease in adsorption capacity per unit area with the increasing extent of activation is generally seen (McKay et al., 1985; Teng and Hsieh, 1998; Mangun et al., 2001), and this has been attributed to the increase in pore diffusion path to reduce the accessibility of inner micropores, or to the enlargement of pores to reduce the overlapping potential for adsorbates. However, the increase in the capacity with the activation level was rarely reported, which may be due to the fact that the analysis of adsorption capacity in terms of q_e/S_{BET} has rarely been used. The difference in the trend may result from the different adsorption potentials associated with the polarity or configuration of the adsorbate relative to the carbon pore walls (Lee and Reucroft, 1999).

To quantitatively comprehend the effect of pore structures on the adsorption capacities, further interpretation of the data with appropriate isotherm models would be necessary. Dubinin and his coworkers (Dubinin, 1980; Dubinin and Stoeckli, 1980; Dubinin, 1982; Dubinin, 1989) have developed equations that simulate equilibrium adsorption of gaseous adsorbates onto different types of microporous carbon adsorbents with energetically homogeneous adsorption sites. These equations are based on the theory of volume filling of the micropores, which describes adsorption as a pore-filling effect, rather than a layer-by-layer adsorption effect (Cal et al., 1994). One of the equations is the DR equation, which has been widely used to describe the adsorption of gases and vapors on microporous solids. The DR equation is written as

$$q_e = q_{DR} \exp\left[-\left(\frac{RT}{E}\right)^2 \ln^2\left(\frac{p_0}{p}\right)\right] \tag{2}$$

where q_{DR} is the ultimate capacity in carbon micropores, E is the characteristic adsorption energy, and p_0 is the saturation pressure of adsorbate at the adsorption temperature T in kelvin.

Equation 2 can be linearized and expressed as

$$\ln q_e = \ln q_{DR} - \left(\frac{RT}{E}\right)^2 \ln^2\left(\frac{p_0}{p}\right) \tag{3}$$

By plotting ln q_e on the ordinate and $\ln^2(p_0/p)$ on the abscissa, and applying a linear regression to the experimental data, q_{DR} and E can be determined from the intercept of the ordinate and the slope of the linear-regression line, respectively. The parameters of the DR equation for the adsorption of acetone and *n*-hexane were determined and the results are shown in Table 3. As expected, the values of the ultimate capacities determined by the DR equation were found to have an order of BW800 > BW700 > BW500, which is identical to that of the carbon porosity. Available pore volume dose be-

Table 3. Parameters of the Dubinin–Radushkevich Equation for Adsorption of Organic Compounds onto Different Carbons

Sample	q_{DR} (mg/g)	E (kJ/mol)	% of Pore Volume
Acetone Adsorption			
BW500	118	42	0.60
BW700	506	27	0.56
BW800	601	25	0.50
n-Hexane Adsorption			
BW500	89	38	0.52
BW700	490	31	0.65
BW800	673	27	0.67

come a limiting factor for adsorption capacity at higher adsorbate concentrations. From Table 3 it can be seen that the adsorption energy decreases with increasing porosity because of pore enlargement to decrease potential overlap between the pore walls (Mangun et al., 1998). Energy consideration is important in maximizing adsorption capacity at low adsorbate concentrations (Cal et al., 1994). Table 3 also shows that the adsorption of acetone onto BW500 has a higher energy than the adsorption of *n*-hexane, while for the adsorption onto BW700 and BW800, the energies are higher for *n*-hexane. The boiling points of acetone and *n*-hexane are 57 and 69°C, respectively. The variation in adsorption energy indicates that, upon activation to widen the pores, a more obvious decrease in adsorption tendency would be observed for adsorption of a more volatile adsorbate, which does not have a strong preference for condensation as a liquid in pores (Mangun et al., 1998).

Using the liquid-phase-specific volumes of 1.26 and 1.52 cm^3/g for acetone and *n*-hexane, respectively, the fractions of pore volume utilized in adsorption were estimated from q_{DR}, and the results are shown in Table 3. As with the trends of q_e/S_{BET} with the activation level, the fraction utilized decreased with the activation level for acetone adsorption, while an increasing trend was found for *n*-hexane. The adsorption of acetone onto BW500 utilized a larger fraction of pore volume than did that of *n*-hexane adsorption, while for the adsorption onto BW700 and BW800 the fractions utilized are larger for *n*-hexane. Again, this can be attributable to the stronger pore-size effect on acetone adsorption. The results in Table 3 strongly indicate that the fraction of pore filling in adsorption is related to the characteristic energy between an adsorbate and an adsorbent, and the energy has been shown to depend on the size of pores in adsorbents (Dubinin, 1989).

Prediction of adsorption isotherms for organic compounds onto different carbons

Because of the heterogeneity in the carbon pore size, as reflected by the pore-size distribution in Figure 2, the DR equation, which is derived based on adsorption on energetically homogeneous sites, cannot provide a satisfactory picture describing the thermodynamics of the adsorption phenomena. In this sense, by applying the DR equation as the local isotherm and correlating the derived energy with the pore size, it is possible to construct a complete description of the heterogeneous adsorption process based on the pore structure of carbon adsorbents.

The slitlike micropores in activated carbons have been thought to be the gaps of molecular dimensions between aromatic sheets and stripes of the carbonaceous structure (Stoeckli, 1990). On the basis of the results from the small-angle X-ray scattering technique, Dubinin (1989) has suggested an inverse proportionality between the slit-pore half-width X and the characteristic adsorption energy E for standard vapor benzene at 20°C, that is

$$X = \frac{k}{E} \tag{4}$$

where k is the structural factor, and its value has been evaluated to be 12.0 kJ·nm/mol for the adsorption of benzene. This relation between the pore size and the adsorption energy shown is used to develop an adsorption model in the present work.

Combining Eqs. 2 and 4, a local isotherm for micropores of a specified size is given as

$$q_X = q_0 \exp\left[-\left(\frac{X}{k}\right)^2 (RT)^2 \ln^2\left(\frac{p_0}{p}\right)\right] \tag{5}$$

where q_X denotes the adsorption capacity in slitlike micropores with a half-width of X, and q_0 is the ultimate capacity. Here we assume that a distribution of micropore size is a fundamental description of heterogeneity of adsorbent surface; that is, the carbon surface comprises many small homogeneous regions that are characterized by the local pore size. Thus, Eq. 5 can be incorporated with the pore-size distribution of an adsorbent to obtain the overall capacity.

Consider the adsorption as a process of volume filling to micropores and let $F(X)$ be the function of micropore-size distribution, then the overall capacity can be given as

$$q_e = \int_{X_{\min}}^{X_{\max}} q_0 \exp\left[-\left(\frac{X}{k}\right)^2 (RT)^2 \ln^2\left(\frac{p_0}{p}\right)\right] \cdot F(X)\, dX \tag{6}$$

where $F(X)\,dX$ represents the micropore volume per gram carbon in micropores having a half-width within X and $X + dX$. The lower limit, $X_{\min}$, is the minumum pore size accessible to adsorbate. Assuming that the adsorbates are spherical and have a close-packed hexagonal arrangement on the carbon surface (Lowell and Shields, 1991), the molecular radiuses of acetone and *n*-hexane were estimated to be 0.344 and 0.426 nm, respectively, and these values were chosen as the lower limits for integration. The upper limit, $X_{\max}$, is the maximum size of micropores that can be obtained in pore-size distribution. As for the value of the ultimate capacity q_0, the liquid-phase densities of the adsorbates, in units of mg/cm^3, are employed, assuming that complete occupancy of the accessible pores is possible.

The argument that the pore-size distribution of carbon determines the adsorption capacity for an adsorbate could be supported, if the adsorption isotherms of the adsorbate onto carbons with different pore structures have been predicted by Eq. 6 using an identical structural factor, k. The adsorption isotherms of BW500 were employed to evaluate the k factors, and values of 5.5 and 11.8 kJ·nm/mol were found to give the best fits for the adsorption of acetone and *n*-hexane, respectively. These values were employed in Eq. 6 to predict the adsorption isotherms of all the carbons, and the results are presented as curves in Figures 3 and 4 to compare with the experimental data. Both figures clearly show that Eq. 6 provides good fits for all the carbons over the entire experimental concentration range. The results demonstrate that the capacity of activated carbon is predominantly controlled by the adsorption energy, which has been considered to be a decreasing function of micropore size shown as Eq. 4.

The pore-size distributions shown in Figure 2 show that the accessible proportion of pore volume for adsorption, that is, the proportion with pore size larger than $X_{\min}$, increases with the extent of activation. However, the preceding interpretation has demonstrated that adsorption energy must be taken into account when assessing the percentage of total pore volume utilized in adsorption. According to Eq. 4, a smaller k value would result in less energy for adsorption at a specified pore size, and would thus render a lower fractional occupancy of pore volume. Figure 7 depicts the pore-size dependence of the fractional occupancy, that is, $\exp[-(RT/E)^2 \ln^2(p_0/p)]$, for k values of 5.5 and 11.8 kJ·nm/mol. It can be seen from the figure that with a smaller k the adsorption occurs mainly in smaller pores and the fractional occupancy decreases more sharply with pore size. This explains why the percentage of total pore volume utilized for acetone adsorption decreases with the extent of porosity development, upon which the pore size increases. On the contrary, the percentage for *n*-hexane adsorption shows an increasing trend with the porosity development. This simply can be ascribed to the increase in the accessible proportion of pore volume, as well as a slower decrease in fractional occupancy with pore size due to a larger k. The difference in the polarity of adsorbate molecules may account for the different k values. The surface of carbon is basically nonpolar and would interact less strongly with polar molecules such as acetone. This would render a much smaller k for the adsorption of acetone, as shown in the present work.

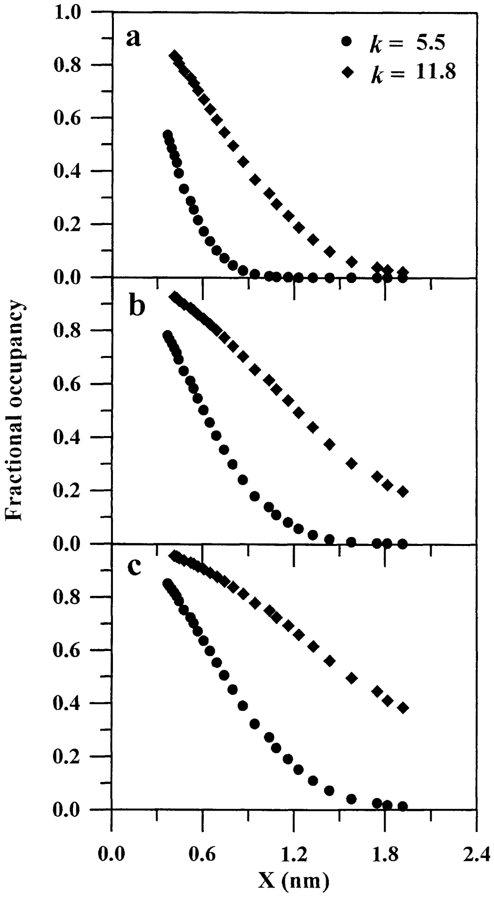

Figure 7. Pore-size dependence of fractional pore-volume occupancy for different *k* values at relative pressures of (a) 0.01, (b) 0.05, and (c) 0.1.

Conclusions

It has been demonstrated on the basis of the present work that the adsorption capacity of activated carbons is controlled by the characteristic adsorption energy that is inversely proportional to the local pore size. The equilibrium capacities for acetone and *n*-hexane adsorption onto KOH-activated carbons have been shown to increase with the extent of porosity development, which was accompanied by an enlargement in pore size. Because of the difference in pore structure, the adsorption capacities did not increase proportionally with the porosity. Using the DR equation as the local isotherm and incorporating it with the pore-size distribution, one is able to simulate the adsorption isotherms for an adsorbate onto different carbons using a structural factor specific to the adsorbate. The structural factor for acetone adsorption was found to be smaller than that for *n*-hexane. The present work has shown that the fractional occupancy of pore volume decreases more sharply with pore size for adsorption characterized by a smaller structural factor. This finding explained satisfactorily why the variations in the percentage of pore volume utilized with the porosity development were different for different adsorbates.

Acknowledgment

Financial support from the National Science Council of Taiwan, through Project NSC 89-2214-E-006-041, is gratefully acknowledged.

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Manuscript received Sept. 6, 2001, and revision received Jan. 15, 2002.