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Adsorption Equilibria of Carbon Dioxide and Ethane on Graphitized Carbon Black[†]

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Adsorption equilibrium data for pure carbon dioxide and ethane on STH-2 graphitized carbon black were measured by a volumetric method. The experiments were carried out over the temperature range (253.2 to 303.2) K and the pressure range (0 to 4) MPa. The relative pressure p/p_0 and adsorption amount n have been adopted to express adsorption isotherms, and the experimental data present a generalized adsorption isotherm over the research range. On the basis of this generalized isotherm, a method of predicting gas adsorption equilibria was explored. The prediction accuracy is satisfactory. In addition, analysis of the BET equation provides proof that the generalized adsorption isotherm $p/p_0 \sim n$ is reasonable.

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1. Introduction

Adsorption of gases on carbonaceous materials has always been an attractive research project because of the different structures and surface properties of various carbonaceous materials. Among these materials, graphitized carbon black (GCB) plays an important role because it could be regarded as having a homogeneous surface. Therefore, graphitized carbon black has often been chosen as a reference material in characterizing the interaction between a molecule and surface carbon atoms.[1] Studies in this field can provide fundamental information on the adsorption potential for other carbonaceous materials. In addition, the adsorption equilibrium prediction models are based on the effects of adsorbate−adsorbent interactions for practical purposes.

An efficient method that is able to predict the adsorption equilibria would be very convenient for adsorption engineering research and applications. The adsorption potential theory developed by Dubinin[2] put forward the concept of the characteristic curve, which could be used to evaluate gas adsorption equilibria using a single experimental adsorption isotherm. Many experimental vapor adsorption equilibrium data on microporous materials, especially on activated carbons, have proved that this method is valid. Attempts to extend the adsorption potential theory to make it applicable to supercritical conditions need more investigation and substantiation. Ruthven[3] provided a similar method, the generalized equilibrium isotherm $K \cdot p \sim n$, to make adsorption predictions. It uses the Henry's law constant K to express the interaction between an adsorbed molecule and the adsorption sites on the surface. This opinion is reasonable for nonpolar systems, experimental data over a wide temperature range are needed for calculation of the Henry's law constant. Molecular simulation is another important prediction approach with assumptions about the solid−fluid interaction, the surface properties, and the pore structure. This method has obtained great success for spherical molecules on carbonaceous materials. The development of molecular simulation requires more support from equilibrium data over a wide experimental range and more improvement in the calculations on nonspherical molecules.[4,5]

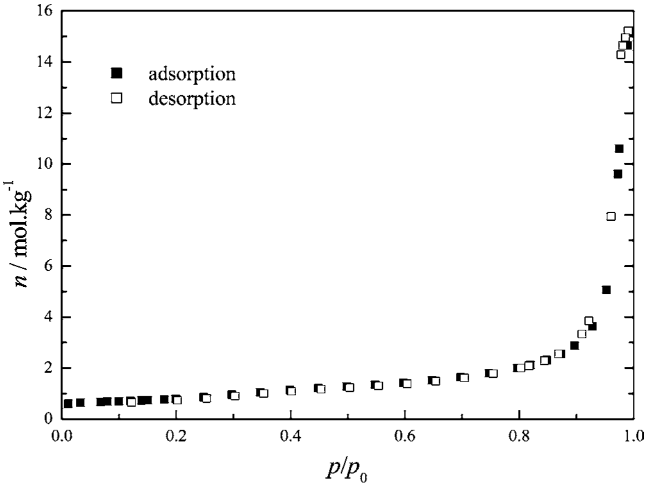

Figure 1. Adsorption and desorption equilibrium data for nitrogen on STH-2 GCB at 77 K: ■, adsorption; □, desorption.

In this paper, adsorption equilibrium data for pure carbon dioxide and ethane on graphitized carbon black over a wide temperature and pressure range are presented. Also, a generalized adsorption isotherm for predicting the adsorption equilibria is proposed.

2. Experimental Section

2.1. Materials. The adsorbent in this work was STH-2 graphitized carbon black supplied by Lanzhou Atech Technologies Company. The STH-2 GCB was in granular form with particle sizes of (0.85 to 1.20) mm. Prior to the experiments, the adsorbent sample was evacuated under vacuum at 473 K for more than 24 h. Nitrogen adsorption and desorption isotherms at 77 K, shown in Figure 1, were measured using a Tristar 3000 static volumetric adsorption analyzer from Micromeritics. No hysteresis loop was observed. On the basis of the linear form of the Brunauer−Emmett−Teller (BET) equation, the specific surface area of STH-2 was calculated as 65.13 $m^2 \cdot g^{-1}$ using experimental data over the relative pressure range $p/p_0 = 0.05$ to 0.35. In addition, gases used in the experiments were helium, ethane, and carbon dioxide. The purities were all higher than 99.99 %.

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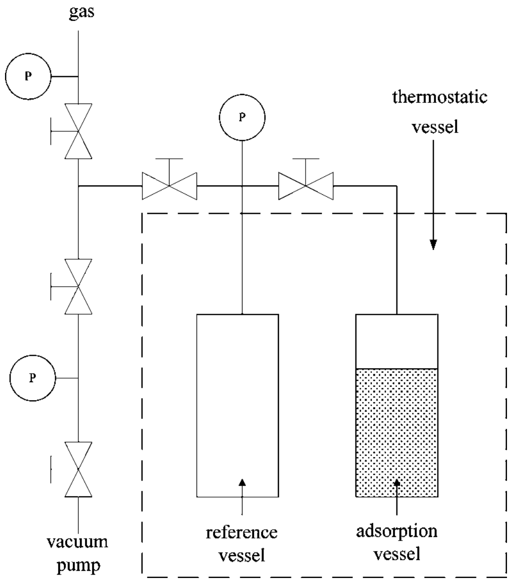

Figure 2. Schematic of the experimental setup.

2.2. Experimental Apparatus. The volumetric method was adopted for measurement of the pure carbon dioxide and ethane adsorption equilibrium data. The apparatus established in our laboratory included an adsorption vessel and a reference vessel with same volume, (82.28 ± 0.05) mL. A schematic of the experimental setup is illustrated in Figure 2. The main part related to the adsorption measurements was kept in a large thermostatic vessel. The experimental temperature range was (253.2 to 303.2) K with an interval of 10 K. The temperature, which was measured using a Pt-100 transducer, could be controlled at the expected value within ± 0.1 K. The equilibrium pressures were measured using a transducer (Keller Druckmesstechnik PAA 200, Switzerland) with a range of (0 to 5) MPa (± 0.00125 MPa). The value of the pressure was monitored and recorded using a computer equipped with a 12-bit A/D converter (Adventech PCI 1710, Taiwan) in order to determine whether the adsorption state approached equilibrium.

2.3. Experimental Procedures. The (19.540 ± 0.001) g adsorbent sample was placed in the adsorption vessel. The adsorption system was degassed under vacuum at 393 K for at least 8 h before each experiment. The dead space was determined using helium adsorption measurements at 298 K over the pressure range (0 to 4) MPa. Helium was initially charged into the reference vessel. The pressure was monitored until it remained constant for 2 h. After that, the gas in the reference vessel was expanded into the adsorption vessel. The change in pressure was recorded by the computer until the adsorption equilibrium was reached. Since it was acceptable to consider the helium to have no adsorption capacity over the experimental range, the mass balance could be used to determine the dead space through the following expression

$$n_1(p_{\text{ref}}, T, V_{\text{ref}}) = n_2(p_{\text{ad}}, T, V_{\text{dead}}) \tag{1}$$

where n_1 and n_2 are the helium amounts before and after adsorption respectively, p_{ref} is the pressure in the reference vessel before adsorption, p_{ad} is the adsorption equilibrium pressure, T is the temperature of the thermostatic vessel, V_{ref} is the volume of the reference vessel, and V_{dead} is the dead space including the free volume in the adsorption vessel and the reference vessel. The volume of the pipes connecting the adsorption vessel and the reference vessel was also considered with temperature calibration. The measurements of helium were repeated with different values of p_{ref} over the pressure range (0 to 5) MPa, and the mean value of V_{dead} was obtained. It should be mentioned that the calculated value of V_{dead} at 298 K in this work was used to determine adsorption equilibria at other temperatures. The recent delicate work of Wang et al.[6] proved that if the temperature range is not small enough, the use of helium to determine the dead space would be related to the temperature. Therefore, the helium calibration and the adsorption measurement should be executed at the same temperature to achieve high accuracy.

After the dead space had been found, the measurement of pure carbon dioxide or pure ethane adsorption equilibria could be carried out using the same procedure. After adsorption had occurred, the difference between the gas amounts before and after adsorption could be used to find the adsorption amount according to the equation

$$n_{\text{ad}} = \frac{n_1(p_{\text{ref}}, T, V_{\text{ref}}) - n_2(p_{\text{ad}}, T, V_{\text{dead}})}{m} \tag{2}$$

where n_{ad} is the amount adsorbed per gram of adsorbent and m is the mass of the adsorbent. The adsorption property was calculated using the Benedict−Webb−Rubin−Starling (BWRS) equation. The average standard deviations in the experiments were 5.2 % for carbon dioxide and 5.4 % for ethane, as obtained from error analysis including the uncertainties in the measurements of pressure, temperature, and adsorbent mass and the uncertainty in the equation of state. It should be mentioned that the estimation of the average standard deviation did not include the systematic error from the change in dead volume with temperature, which may introduce an additional uncertainty of about (1 to 2) %.

With the assumption that the molecular area of carbon dioxide is 0.206 nm^2, the experimental data for carbon dioxide at 273.2 K could be used determine another value of the specific surface area, namely, 63.81 $\text{m}^2 \cdot \text{g}^{-1}$.[7] This result may be consistent with the value from nitrogen adsorption at 77 K, since there were two different adsorption systems and measurement procedures.

3. Results and Discussion

3.1. CO_2 and C_2H_6 Adsorption Isotherms. Experimental adsorption isotherms for CO_2 and C_2H_6 on STH-2 GCB are presented in Figures 3 and 4, respectively. The adsorption data are given in detail in Tables 1 to 4.

To express the adsorption equilibria of CO_2 and C_2H_6 on STH-2 GCB, the BET equation in the following form was used:

$$\frac{n}{n_{\text{m}}} = \frac{C\dfrac{p}{p_0}}{\left[1 - \dfrac{p}{p_0}\right]\cdot\left[1 - \dfrac{p}{p_0} + C\dfrac{p}{p_0}\right]} \tag{3}$$

where n is the amount adsorbed at equilibrium, n_{m} is the adsorption amount at full monolayer coverage, p and p_0 are equilibrium and saturated pressures, respectively, and C is a parameter related to the adsorption interaction. For thermodynamic considerations, the pressure should be changed to the fugacity through the BWRS equation in the following discussion.

Through the linear form of the BET equation, values of the parameters n_{m} and C could be calculated from the experimental

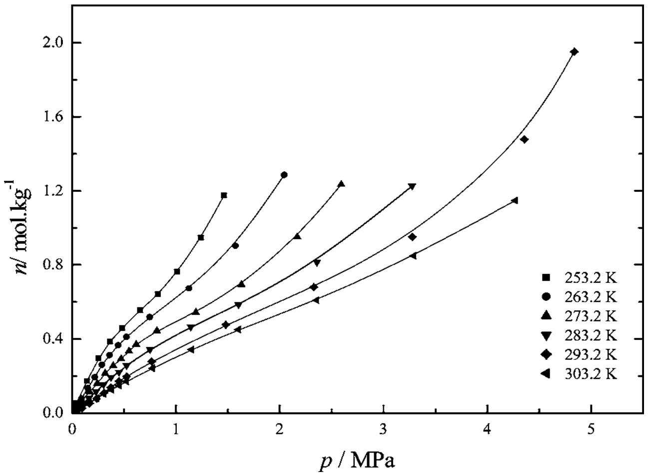

Figure 3. Adsorption equilibrium data for carbon dioxide on STH-2 GCB: ■, 253.2 K; ●, 263.2 K; ▲, 273.2 K; ▼, 283.2 K; ◆, 293.2 K; left-pointing triangles, 303.2 K.

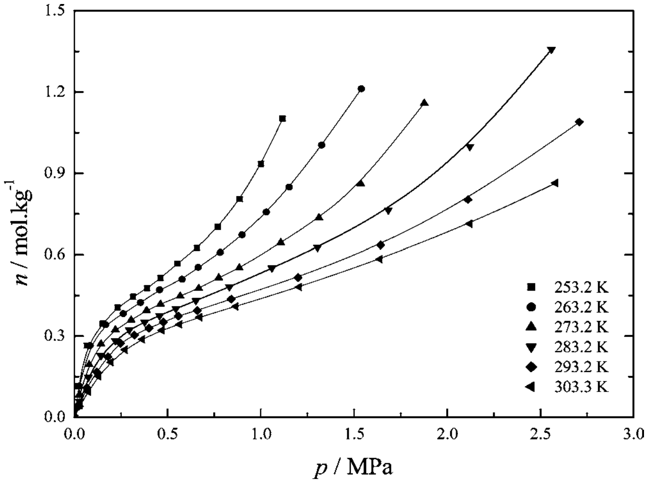

Figure 4. Adsorption equilibrium data for ethane on STH-2 GCB: ■, 253.2 K; ●, 263.2 K; ▲, 273.2 K; ▼, 283.2 K; ◆, 293.2 K; left-pointing triangles, 303.2 K.

Table 1. Adsorption Equilibrium Data for Carbon Dioxide on STH-2 GCB at (253.2, 263.2, and 273.2) K

T = 253.2 K		T = 263.2 K		T = 273.2 K	
p	n	p	n	p	n
MPa	mol·kg^{-1}	MPa	mol·kg^{-1}	MPa	mol·kg^{-1}
0.0425	0.0527	0.0350	0.0335	0.0350	0.0275
0.1425	0.1727	0.0900	0.0779	0.0925	0.0659
0.2550	0.2951	0.1500	0.1345	0.1675	0.1147
0.3650	0.3861	0.2175	0.1941	0.2425	0.1591
0.4825	0.4596	0.2875	0.2606	0.3175	0.2144
0.6550	0.5549	0.3600	0.3130	0.3950	0.2559
0.8250	0.6416	0.4450	0.3650	0.4725	0.2928
1.0100	0.7628	0.5250	0.4109	0.5475	0.3349
1.2400	0.9467	0.7475	0.5176	0.6175	0.3707
1.4625	1.1765	1.1250	0.6730	0.8175	0.4445
		1.5725	0.9030	1.1925	0.5435
		2.0425	1.2863	1.6300	0.6939
				2.1700	0.9516
				2.5925	1.2341

Table 2. Adsorption Equilibrium Data for Carbon Dioxide on STH-2 GCB at (283.2, 293.2, and 303.2) K

T = 283.2 K		T = 293.2 K		T = 303.2 K	
p	n	p	n	p	n
MPa	mol·kg^{-1}	MPa	mol·kg^{-1}	MPa	mol·kg^{-1}
0.0400	0.0185	0.0375	0.0084	0.0425	0.0178
0.0950	0.0456	0.0950	0.0261	0.0975	0.0344
0.1600	0.0803	0.1675	0.0523	0.1625	0.0578
0.2300	0.1167	0.2350	0.0774	0.2375	0.0795
0.3025	0.1542	0.3050	0.1082	0.3050	0.1040
0.3750	0.1922	0.3725	0.1386	0.3800	0.1263
0.4500	0.2216	0.4500	0.1721	0.4525	0.1482
0.5250	0.2565	0.5250	0.1959	0.5250	0.1703
0.7500	0.3437	0.7650	0.2781	0.7725	0.2411
1.1425	0.4663	1.4800	0.4767	1.1500	0.3416
1.6025	0.5859	2.3275	0.6798	1.6025	0.4503
2.3575	0.8146	3.2775	0.9516	2.3550	0.6093
		4.3575	1.4783	3.2925	0.8486
				4.2700	1.1474

Table 3. Adsorption Equilibrium Data for Ethane on STH-2 GCB at (253.2, 263.2, and 273.2) K

T = 253.2 K		T = 263.2 K		T = 273.2 K	
p	n	p	n	p	n
MPa	mol·kg^{-1}	MPa	mol·kg^{-1}	MPa	mol·kg^{-1}
0.0225	0.1148	0.0250	0.1161	0.0275	0.0835
0.0675	0.2652	0.0850	0.2656	0.0800	0.1948
0.1525	0.3465	0.1675	0.3425	0.1425	0.2713
0.2325	0.4059	0.2625	0.3832	0.2200	0.3237
0.3150	0.4452	0.3550	0.4243	0.3050	0.3592
0.3900	0.4768	0.4575	0.4710	0.3875	0.3949
0.4625	0.5145	0.5775	0.5098	0.4625	0.4180
0.5525	0.5677	0.6650	0.5541	0.5675	0.4476
0.6575	0.6245	0.7825	0.6086	0.6675	0.4764
0.7700	0.7029	0.9000	0.6732	0.7750	0.5156
0.8875	0.8048	1.0300	0.7570	0.8850	0.5515
1.0025	0.9358	1.1525	0.8498	1.1075	0.6439
1.1175	1.1024	1.3275	1.0047	1.3125	0.7358
		1.5400	1.2124	1.5375	0.8614
				1.8775	1.1591

Table 4. Adsorption Equilibrium Data for Ethane on STH-2 GCB at (283.2, 293.2, and 303.2) K

T = 283.2 K		T = 293.2 K		T = 303.2 K	
p	n	p	n	p	n
MPa	mol·kg^{-1}	MPa	mol·kg^{-1}	MPa	mol·kg^{-1}
0.0300	0.0579	0.0250	0.0412	0.0300	0.0454
0.0750	0.1486	0.0675	0.1098	0.0750	0.0948
0.1425	0.2281	0.1200	0.1680	0.1300	0.1519
0.2200	0.2831	0.1800	0.2246	0.1975	0.2041
0.2950	0.3234	0.2475	0.2747	0.2725	0.2502
0.3775	0.3521	0.3225	0.3041	0.3650	0.2885
0.4575	0.3754	0.4000	0.3300	0.4700	0.3220
0.5450	0.4019	0.4775	0.3516	0.5650	0.3438
0.6525	0.4312	0.5575	0.3745	0.6725	0.3702
0.8325	0.4819	0.6600	0.3950	0.8675	0.4098
1.0600	0.5510	0.8425	0.4373	1.2075	0.4816
1.3050	0.6273	1.2000	0.5166	1.6400	0.5843
1.6850	0.7639	1.6425	0.6356	2.1225	0.7140
2.1225	0.9992	2.1125	0.8030	2.5825	0.8642
2.5600	1.3576	2.7100	1.0894		

data between relative pressures of about 0.05 and 0.35. The results are shown in Table 5. For both CO_2 and C_2H_6, the values of C decreased with increasing temperature, indicating the effect of the equilibrium temperature on the adsorbate−adsorbent interactions. The value of n_m remained almost constant over the experimental temperature range, which is consistent with the viewpoint of Hoory and Prausnitz[8] and Findenegg.[9] The value of n_m at 303.2 K was a little higher, perhaps because this temperature is close to the critical temperatures of CO_2 (304.5 K) and C_2H_6 (305.4 K). The adsorbate molecule is more compressible under that condition.

3.2. The $p/p_0 \sim n$ Generalized Isotherm. Figures 5 and 6 show adsorption isotherms for CO_2 and C_2H_6, respectively, using the relationship between the adsorption equilibrium amount n and the relative pressure p/p_0. It can be observed that the

Table 5. Parameters of the BET Equation

	CO_2		C_2H_6	
$\frac{T}{K}$	$\frac{n_m}{mol \cdot kg^{-1}}$	C	$\frac{n_m}{mol \cdot kg^{-1}}$	C
253.2	0.512	7.21	0.363	35.50
263.2	0.533	6.55	0.369	32.47
273.2	0.537	5.82	0.369	29.55
283.3	0.545	5.31	0.381	23.11
293.2	0.545	5.30	0.389	23.00
303.2	0.580	4.99	0.408	20.06

experimental data present a distinct single curve for each adsorbate. This means that if the gas adsorption isotherms are plotted as n versus p/p_0, we can get a single generalized isotherm to express all of the experimental data. This opinion is supported by the work of Lee et al.,[10] who obtained a similar generalized isotherm for organic vapors on mesoporous silica gel MCM-48.

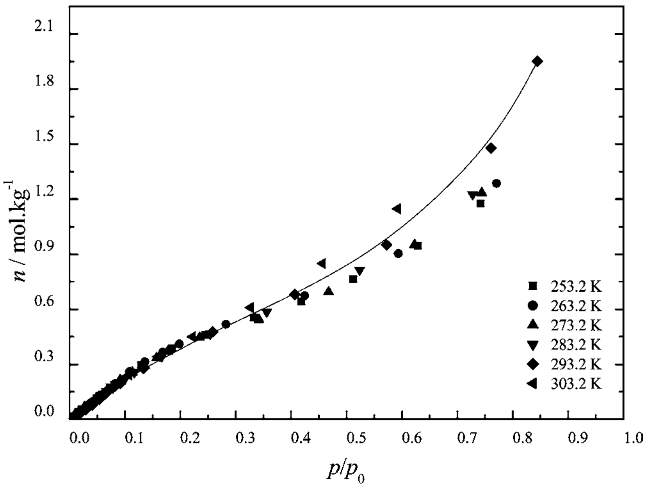

Figure 5. Generalized adsorption isotherm for carbon dioxide on STH-2 GCB: ■, 253.2 K; ●, 263.2 K; ▲, 273.2 K; ▼, 283.2 K; ◆, 293.2 K; left-pointing triangles, 303.2 K.

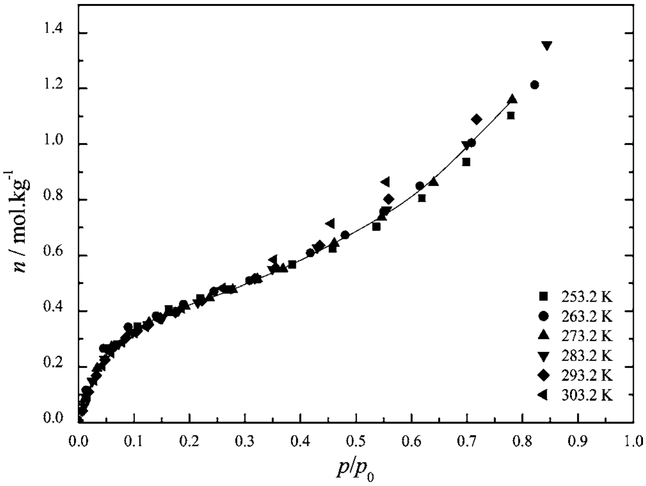

Figure 6. Generalized adsorption isotherm for ethane on STH-2 GCB: ■, 253.2 K; ●, 263.2 K; ▲, 273.2 K; ▼, 283.2 K; ◆, 293.2 K; left-pointing triangles, 303.2 K.

The following analysis demonstrates that the generalized isotherm $n \sim p/p_0$ is reasonable in some cases. The BET equation can also be expressed as

$$\frac{n}{n_m} = f\left(C, \frac{p}{p_0}\right) \tag{4}$$

Thus, n/n_m is a function of the parameter C and the relative pressure p/p_0. Differentiation of n/n_m with respect to C gives

$$\frac{d(n/n_m)}{dC} = \frac{p/p_0}{[1 - (p/p_0) + C \cdot (p/p_0)]^2} \tag{5}$$

The value of $d(n/n_m)/dC$ as a function of C and p/p_0 is plotted in Figure 7. It can be seen that most values of $d(n/n_m)/dC$ approach zero except when the value of C is much lower than 20. Consequently, variation of C and p/p_0 has little effect on $d(n/n_m)/dC$ when the value of C is large enough. Values of $d(n/n_m)/dC$ averaged over the whole experimental relative pressure range (0.001 to 1) for various constant values of C are presented in Table 6. It is important to note that when C is larger than 15, the mean value of $d(n/n_m)/dC$ is less than 0.01. Thus, different values of C would not change the value of n/n_m greatly. On the other hand, n/n_m is sensitive to changes in C when C is less than 5.

The ranges of parameter C for CO_2 and C_2H_6 were 5 to 8 and 20 to 40, respectively, in this work. In addition, the value of n_m could be assumed to be constant over the research temperature range. Therefore, the adsorption equilibrium amount n could be determined from eq 3 at a certain relative pressure p/p_0. The temperature 293.2 K was selected as the reference temperature T_{ref}, and the corresponding adsorption equilibrium data was used to establish the generalized adsorption isotherm. Adsorption equilibria at other temperatures were then calculated from the generalized adsorption isotherm. The prediction uncertainty was determined by the mean relative deviation δ at the given temperature:

$$\delta = \sqrt{\frac{1}{N}\sum_{i=1}^{N}\left(\frac{n_i - n_{calcd,i}}{n_i}\right)^2} \tag{6}$$

In Table 7, the values of δ for CO_2 and C_2H_6 are 0.099 and 0.062, respectively. The prediction results are satisfactory and demonstrate that this generalized adsorption isotherm can be applied to the prediction of pure gas adsorption equilibria on GCB. The prediction of CO_2 is not more accurate than C_2H_6 because its C value is lower (close to 5).

Since the BET equation can be used not only for adsorbents with homogeneous surfaces but also for some mesoporous adsorbents, we attempted to extend this method to estimate gas adsorption equilibria on various kinds of adsorbents reported in the literature.[11−14] The results are also given in Table 7. It is interesting that although the adsorbents include nonporous, mesoporous, and even microporous solids, the mean relative deviations of the prediction are acceptable (most of the values of δ are less than 10 %).

The relative pressure in this method, which is related to the equilibrium temperature, reflects the effect of temperature on the adsorption equilibria. In comparison with the generalized adsorption isotherm $K \cdot p \sim n$ suggested by Ruthven,[3] the relationship between p/p_0 and n is more convenient for prediction because it does not require calculation of the Henry's law constant K. It should be mentioned that the generalized adsorption isotherm $p/p_0 \sim n$ is not suitable for supercritical

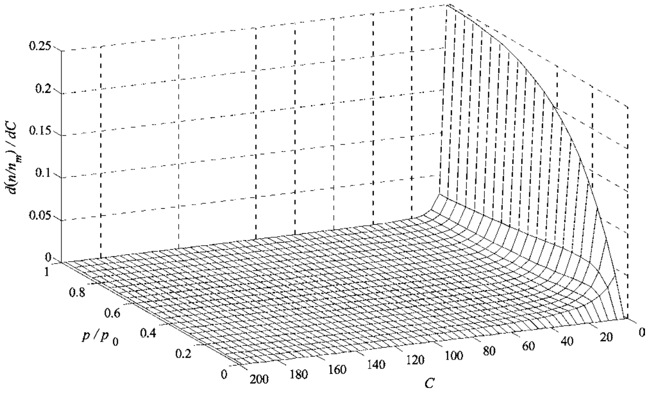

Figure 7. Effects of the parameter C and the relative pressure p/p_0 on $d(n/n_m)/dC$.

Table 6. Values of $d(n/n_m)/dC$ Averaged over the Whole Range of Relative Pressure for Different Values of C

C	2	3	5	10	15
$d(n/n_m)/dC$	0.2052	0.1140	0.0531	0.0169	0.0088
C	20	30	40	50	100
$d(n/n_m)/dC$	0.0053	0.0026	0.0015	0.0009	0.0002

Table 7. Predicted Results of Gas Adsorption Equilibria on Adsorbents

gas	T/K	T_{ref}/K	δ	adsorbent
CO_2	253.2 to 303.2	293.2	0.099	GCB STH-2 GCB
C_2H_6	253.2 to 303.2	293.2	0.062	GCB STH-2 GCB
CCl_4[a]	278.2 to 296	288.4	0.040	GCB P-33
$CFCl_3$[a]	238.2 to 248.2	238.2	0.078	GCB P-33
$CFCl_3$[a]	260.2 to 286.2	273.2	0.092	GCB P-33
$CHCl_3$[a]	223.2 to 231.2	223.2	0.026	GCB P-33
$CHCl_3$[a]	268.2 to 288.3	268.2	0.072	GCB P-33
CCl_4[b]	300.2 to 330.2	310.2	0.091	Sorbonorit B4
$CHCl_3$[b]	300.2 to 330.2	310.2	0.091	Sorbonorit B4
CO_2[c]	298 to 303	298	0.029	activated carbon F30/470
C_6H_6[d]	303.2 to 333.2	313.2	0.083	Sorbonorit B4

[a] Using adsorption data from ref 11. [b] Using adsorption data from ref 12. [c] Using adsorption data from ref 13. [d] Using adsorption data from ref 14.

gas adsorption, since the saturated pressure p_0 does not exist under supercritical conditions.

4. Conclusions

In this work, carbon dioxide and ethane adsorption equilibrium data on STH-2 GCB were measured using a volumetric method over the temperature range (253.2 to 303.2) K and the pressure range (0 to 4) MPa. The relationship between the relative pressure p/p_0 and the adsorption equilibrium amount n establishes a generalized adsorption isotherm. From analysis of the BET equation, the generalized adsorption isotherm $p/p_0 \sim n$ is proved reasonable under the condition that the parameter C is not very small. On the basis of this generalized adsorption isotherm, a method for predicting adsorption equilibrium data has been proposed. The prediction results have satisfactory accuracy.

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