Adsorption of Organic Solvent Vapors on Hydrophobic Y-Type Zeolite

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Experimental isotherms and prediction results for adsorption of benzene, toluene, dichloromethane and 1,1-dichloro-1-fluoroethane on hydrophobic Y-type zeolite are reported. Isotherm shows the type-V shape according to the classification by Brunauer et al. A simple thermodynamic method is employed to predict the experimental equilibrium data at various temperatures simultaneously. This plain method is based on the assumption that the value of the isosteric heat of adsorption does not depend on temperature for a certain surface loading. The Clausius–Clapeyron equation was used to calculate the isosteric heat of adsorption. To apply the method, only two sets of the experimental isotherm data at two different temperatures are needed. The Clausius–Clapeyron equation with two isotherms provided simple and reliable prediction of adsorption equilibrium relationships at various temperatures. Our results with this method showed that the predicted value agrees well with the experimental data in the range of temperatures for the system tested.

Introduction

The emission control of volatile organic compounds (VOCs) by adsorption process has attracted special interest as a means of protecting the environment and human health from air pollution. In general, it has been recognized that activated carbon is the most suitable adsorbent for this application (Ruhl, 1993). However, Blocki (1993) pointed out that the applications of activated carbon present some disadvantages in that they are flammable, are difficult-to-regenerate high-boiling solvents, they promote polymerization or oxidation of some solvents to toxic or insoluble compounds, and require humidity control. Therefore, it is necessary for the new type of adsorbent to replace the activated carbon. As a result, hydrophobic zeolite is now considered an alternative adsorbent since it has good properties such as thermal stability and hydrophobicity (Blocki, 1993; Takeuchi et al., 1995; Tsai et al., 1996; Yen and Teng, 1996).

Zeolites are inorganic materials that have a crystalline structure and fixed-size pores. The homogeneous pore size prevents molecules larger than a certain size from entering the lattice, so zeolites are sometimes called molecular sieves, which allows them to adsorb selectively. The nonflammable, thermal-stable, and hydrophobic characteristics of zeolites can also play an important role in adsorption. The thermal stability and hydrophobicity of zeolite increase with the Si/Al ratio in the zeolite framework. Synthetic hydrophobic zeolite, a pure crystalline silica molecular sieve, is nonflammable and capable of withstanding temperatures as high as 850°C (Deng and Lin, 1995). Furthermore, hydrophobic zeolite has a low affinity for water, which is a useful physical property. Hussey and Gupta (1996) reported that up to 90% relative humidity can be handled with little adverse effect on the capacity of hydrophobic zeolite. Also, Takeuchi et al. (1995) reported that the presence of water vapor was found to reduce the amount of adsorption of the solvents–hydrophobic zeolite system, but water vapor showed no effect on the adsorption kinetics.

To design adsorption facilities, thermodynamic data on the adsorption equilibria are essential (Yang, 1987). For practical applications, adsorption equilibria must be known over a wide range of the operation temperature. However, there appears to be relatively little information in the literature concerning adsorption of organic solvents on hydrophobic zeolite at various temperatures. In most adsorption studies, experimental data obtained were fitted to a specific isotherm equation that included the parameters as a function of temperature (Hwang

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et al., 1997; Olivier et al., 1994; Cochran et al., 1985). However, it may not be easy to accurately determine an appropriate set of isotherm parameters that can represent all of the experimental data at different temperatures. This is because there must be a certain temperature dependency in the isotherm parameters, and at least three sets of isotherm data are needed for the temperature range of interest. In addition, its numerical procedure is somewhat complex.

What we present in this article are (1) adsorption equilibrium data of organic solvent vapors (benzene, toluene, dichloromethane, and 1,1-dichloro-1-fluoroethane) on hydrophobic Y-type zeolite at various temperatures; and (2) the applicability of a simple thermodynamic method for predicting the adsorption equilibrium relationships of the solvent vapors–hydrophobic zeolite system. This simple method is based on the idea that the value of the isosteric heat of adsorption is not dependent on adsorption temperature for a certain surface loading. Our results using this method showed that the predicted value is in good agreement with the experimental data within the temperature range for the system tested.

Adsorption Equilibrium Theory

For many decades, numerous researchers have considered adsorption equilibrium from the thermodynamic perspective and developed a number of theories for the equilibrium isotherm based on various assumptions concerning the nature of adsorbed phase (Ruthven, 1984). Adsorption equilibrium is established after the gas has been in with the adsorbed surface for a long time, and can be represented in the general form:

$$f(N, P, T) = 0. \tag{1}$$

In this equation, N is the amount adsorbed, P is the equilibrium pressure, and T is the temperature. For the isothermal condition, Eq. 1 can be represented by the adsorption isotherm:

$$N = f(P)_T \quad \text{and} \quad P = f^{-1}(N)_T. \tag{2}$$

The adsorption isotherm, Eq. 2, is most frequently used in studies of adsorption equilibrium, and is represented by mathematical equations such as the Langmuir, the Freundlich, the Sips, the Toth, and the Unilan (Yang, 1989; Valenzuela and Myers, 1989). Equation 1 can also take the following form and is called the adsorption isostere (Young and Crowell, 1962):

$$P = f(T)_N \tag{3}$$

However, the adsorption isostere cannot be measured directly because it is impractical to hold N constant.

It has been generally known that adsorption is accompanied by evolution of heat, since adsorbate molecules are more stabilized on the adsorbent surface than in the bulk phase (Suzuki, 1990). In addition, the amount of heat evolution by unit adsorption depends on the system adopted. Thus, information concerning the magnitude of the heat of adsorption and its transition with surface coverage can provide useful information concerning the nature of the surface and the adsorbed phase (Ross and Olivier, 1964; Suzuki, 1990; Young and Crowell, 1962). The isosteric heat of adsorption, q_{st} (sometimes denoted as $-\Delta H$), is defined by an equation of the Clausius–Clapeyron type (Hill, 1949):

$$\frac{q_{st}}{RT^2} = \left[\frac{\partial \ln P}{\partial T}\right]_N. \tag{4}$$

Equation 4, when integrated, allows one to calculate q_{st} from adsorption isotherms obtained experimentally at two or more temperatures. If experimental isotherms are measured at two temperatures, the value of q_{st} is given by (Ross and Olivier, 1964; Suzuki, 1990; Valenzuela and Myers, 1989):

$$q_{st} = R\left[\frac{\ln P_1 - \ln P_2}{1/T_2 - 1/T_1}\right]_N \tag{5}$$

where P_1 and P_2 are the equilibrium pressures at temperatures T_1 and T_2, respectively, when the amount adsorbed is N.

As stated by Hill (1949), when the surfaces are energetically homogeneous and there is no interaction between adsorbed molecules, the isosteric heat of adsorption is independent of the amount adsorbed. However, if different levels of surface energy exist and the interactions between adsorbed molecules cannot be neglected, the isosteric heat of adsorption varies with the surface coverage (Hill, 1949; Suzuki, 1990).

Experimental Studies

A commercial hydrophobic Y-type zeolite, designated as DAY by Degussa Co., was chosen as an adsorbent, and its 20- and 30-mesh ratio was used after crushing. Prior to measurement, the sample was kept in a drying vacuum oven at 423 K for more than 24 h to remove impurities. The specific surface area of the sample was obtained on an automatic volumetric sorption analyzer (Micromeritics, ASAP-2000) using nitrogen adsorption at 77 K. The measured and supplied properties of zeolite used are tabulated in Table 1. The adsorbates investigated were benzene, toluene, dichloromethane (DCM), and 1,1-dichloro-1-fluoroethane (HCFC-141b). The purity and manufacturer of each adsorbate are benzene: 99.9% (J.T. Baker); toluene: 99.9% (J.T. Baker); dichloromethane: 99.0% (Junsei); 1,1-dichloro-1-fluoroethane: 99.8% (Elf Atochem).

Table 1. Physical Properties of Hydrophobic Y-Type Zeolite (DAY)

Property	Value
Bulk density, $kg \cdot m^{-3}$	502
Specific surface area*, $m^2 \cdot g^{-1}$	800
Specific surface area**, $m^2 \cdot g^{-1}$	566
Micropore area**, $m^2 \cdot g^{-1}$	526
Micropore volume*, $cm^3 \cdot kg^{-1}$	300
Micropore volume**, $cm^3 \cdot kg^{-1}$	245
Pore distribution**, %	
≤ 50 Å	35.34
50–500 Å	43.15
≥ 500 Å	21.51
Average pore diameter**, Å	73.89

*From manufacturer's report.
**From BET experiment by ASAP-2000.

We used a static-volumetric-type apparatus. The total quantity of gas admitted to the system and the amount of gas remaining in the vapor after adsorption equilibrium was established were determined by appropriate *P-V-T* measurements. The pressure measurements of the system were made by a baratron absolute pressure transducer (MKS type 690A13TRA) with a high accuracy signal conditioner (MKS type 270D), a pressure range of 0–133.33 kPa, and a reading accuracy of 0.05% in the usable measurement range. During adsorption, the adsorption cell was placed in a water bath and temperature was kept constant within ± 0.02 K by the refrigerating circulator (Haake type F3). The experimental temperature was measured with a T-type thermocouple converter. To eliminate any trace of pollutants, the activated carbon was kept in a drying oven for 24 h at 423 K. Its mass was determined with an accuracy of ± 10 μg and was introduced into the adsorption cell. Prior to each isotherm measurement, the charged activated carbon was regenerated at 473 K under a high vacuum for 12 h. A combination oil diffusion pump and mechanical vacuum pump (Edward type Diffstak 63/150 M) provided a vacuum down to 10^{-3} Pa, and the evacuation was monitored by both an ion gauge and a convectron gauge with a vacuum-gauge controller (Granville-Phillips type 307). The volume of the adsorption cell was determined by the expansion of helium gas at the experimental temperature. Details of the equipment and the operating procedures used are described in an article by Yun and Choi (1997).

Experimental Results and Correlation

The adsorption equilibria for benzene, toluene, dichloromethane (DCM), and 1,1-dichloro-1-fluoroethane (HCFC-141b) on a commercially available hydrophobic Y-type zeolite (DAY) were obtained by a static volumetric apparatus. Temperatures ranging from 288 K to 373 K were used to measure the isotherms for pure vapors at pressures up to 6 kPa for benzene, 2 kPa for toluene, 30 kPa for dichloromethane, and 60 kPa for 1,1-dichloro-1-fluoroethane, respectively. Typical adsorption isotherms of various organic vapors on hydrophobic Y-type zeolite are shown in Figure 1. In this figure, the isotherms show S-shaped isotherms in the low pressure range and they can be classified as type V according to the classification by Brunauer et al. (1940). It has been reported that type V isotherms describe the behavior of adsorptive systems with large intermolecular attraction effects, such as sorption of water vapors on activated carbon, or sorption of phosphorous vapors on zeolite NaX (Kruglov and Aris, 1995).

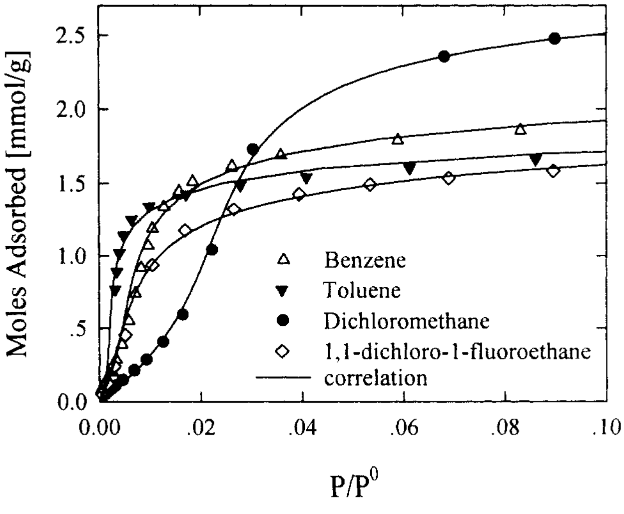

Figure 1. Adsorption isotherms of organic solvents on hydrophobic Y-type zeolite at 323 K.

Generally, the equilibrium data obtained should be represented by a specific mathematical model (i.e., an isotherm equation). This allows one to obtain physical parameters of a model used over a wide range of pressures and temperatures. Furthermore, the adsorption equilibrium model is essential in calculating the breakthrough curve in the adsorptive separation process simulation (Yun et al., 1996). However, the adsorption system that has S-shaped isotherms cannot be expressed by simple equations (such as the Langmuir, the Freundlich, the Sips, the Toth, or the Unilan) because of a lack of flexibility in their mathematical formulas. In this study, the following general adsorption isotherm equation, which was proposed by Martinez and Basmadjian (1997), was used to correlate the experimental data:

$$P = M_0 e^{-Q/kT} \frac{\theta}{(1-\theta)^r}, \qquad \theta = \frac{N}{N^\infty} \tag{6}$$

and

$$Q = \chi - rW\theta, \tag{7}$$

where N^∞ is the maximum moles adsorbed, Q is the heat of adsorption and r is the ratio of the size of the adsorbed phase to the maximum moles adsorbed. All of which means that $M_0 e^{-Q/kT}$ is equal to the slope of the isotherm at low pressure and Q is equal to the minimum energy required to desorb all molecules from their lowest energy state in the adsorbed phase χ (Martinez and Basmadjian, 1997).

Equation 6 contains five adjustable parameters (N^∞, M_0, χ/k, W/k, and r), which allows it to be sufficiently flexible to fit our experimental data. In order to find the isotherm parameters for each adsorption system, we used the so-called Nelder-Mead simplex pattern search algorithm (Riggs, 1988). Table 2 contains the parameters of the general isotherm equation for benzene, toluene, dichloromethane, and 1,1-dichloro-1-fluoroethane on a hydrophobic Y-type zeolite. In this work, a deviation parameter (DP) on equilibrium pressure was utilized to compare the correlation results with experimental data:

$$\mathrm{DP} = \frac{100}{k} \sum_i^k \left| \frac{P_i^{\mathrm{obs}} - P_i^{\mathrm{cal}}}{P_i^{\mathrm{obs}}} \right|, \qquad k = \text{number of data}. \tag{8}$$

As discussed in the previous section, the isosteric heat of adsorption can be calculated by the Clausius–Clapeyron equation of adsorption. The isosteric heat of adsorption for all the vapors studied are plotted as a function of the moles adsorbed in Figure 2. The isosteric heat of adsorption, which is a measure of the interactions between adsorbate molecules and adsorbent lattice atoms, may be used as a measure of the energetic heterogeneity of a surface. As shown in Figure 2, the isosteric heat of adsorption varies with the surface load-

Table 2. General Adsorption Isotherm Parameters of Organic Solvent Vapors on Hydrophobic Y-Type Zeolite

Temp. K	N^∞ mmol/g	M_0 kPa	χ/k K	W/k K	r	DP %
			Benzene			
288.15	3.112	2.275	348.6	−441.0	18.56	14.7
303.15	2.711	2.401	362.7	−479.8	8.962	12.3
313.15	2.778	2.752	257.2	−481.0	8.875	10.6
323.15	2.614	2.781	205.4	−497.1	5.826	8.63
333.15	2.730	5.839	242.0	−505.0	6.908	5.95
343.15	2.880	6.147	104.8	−496.3	8.237	4.35
357.85	2.723	6.396	24.15	−493.0	5.216	4.14
372.65	2.678	11.77	29.01	−517.8	4.970	2.88
			Toluene			
303.15	2.546	0.188	556.8	−408.8	14.21	10.4
313.15	2.329	0.203	114.4	−487.0	10.32	13.9
323.15	2.327	1.429	409.9	−509.9	11.38	9.46
333.15	2.167	1.730	383.9	−543.3	7.818	6.94
343.15	2.196	1.672	248.1	−538.6	7.655	6.25
			Dichloromethane			
283.15	3.418	18.05	472.8	−587.8	3.586	6.06
303.15	3.177	32.13	444.2	−650.0	2.373	6.47
323.15	2.911	46.47	413.9	−692.0	1.227	7.75
			1,1-Dichloro-1-Fluoroethane			
283.15	2.747	3.405	359.1	−389.3	9.319	10.1
303.15	2.777	6.168	230.3	−401.2	9.355	6.59
323.15	2.795	14.25	211.6	−409.4	10.26	7.86
343.15	2.644	32.21	165.2	−456.9	11.10	6.99
363.15	2.594	70.69	219.9	−472.1	11.06	5.84

ing for all sample adsorbates, suggesting that adsorption occurs on an energetically heterogeneous surface. At the zero coverage limit, the values of the isosteric heat of adsorption are 32.50 kJ · mol^{-1} for benzene, 72.81 kJ · mol^{-1} for toluene, 28.49 kJ · mol^{-1} for dichloromethane, and 40.61 kJ · mol^{-1} for 1,1-dichloro-1-fluoroethane, respectively.

Prediction with the Isosteric Heat of Adsorption

For practical use, the experimental equilibrium data obtained should be represented by a mathematical isotherm equation that includes the adjustable parameters as a function of temperature. As noted by Do and Do (1997), the choice of isotherm equation for a specific adsorption system has been a topic of great interest for many decades to researchers working in the adsorption area. As a result, many theories and empirical methods have been presented ranging from the traditional kinetics approach to molecular simulations. In any case, however, it still may not be easy to determine a set of isotherm parameters that can represent all of the experimental data at different temperatures with good accuracy. This is because there has to be a certain temperature dependency in the isotherm parameters themselves, and at least three sets of isotherm data are required for the temperature range of interest. Furthermore, the numerical optimization procedure is somewhat complex.

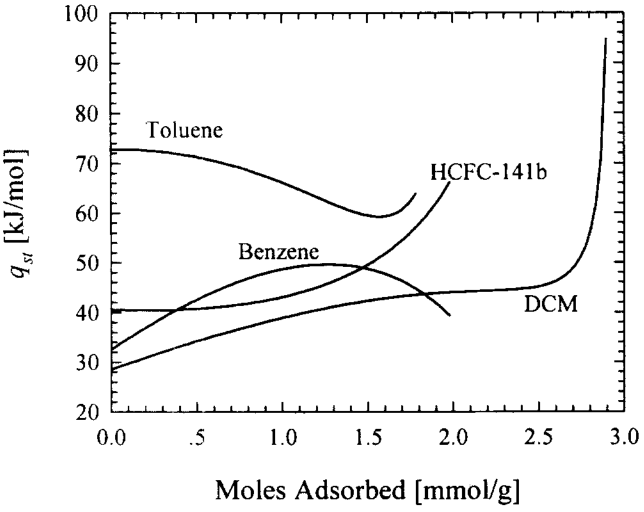

Figure 2. Isosteric heat curves for organic solvents by hydrophobic Y-type zeolite.

As a useful thermodynamic property, the isosteric heat of adsorption generally has been used to characterize the adsorbent surface. Besides, this thermodynamic property can be used to verify the reliability of experimental isotherm data. In their adsorption equilibrium database, Valenzuela and Myers (1989) introduced a useful method that can estimate the temperature coefficient of amount adsorbed by the Clausius–Clapeyron equation. This estimation is based on the approach that the isosteric heat of adsorption is independent of temperature. By introducing this approach one can obtain the following useful relation:

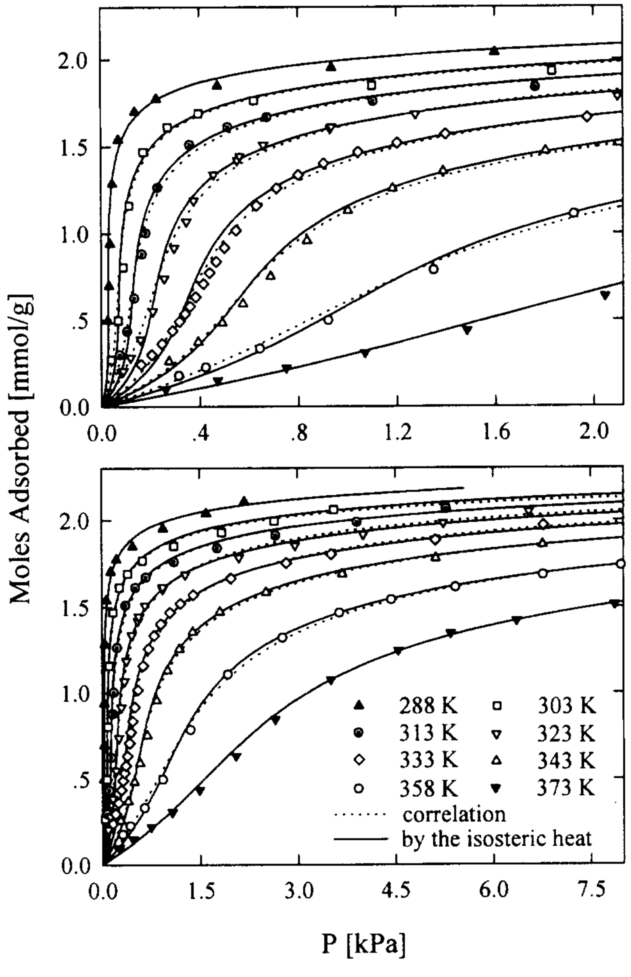

Figure 3. Adsorption isotherms for benzene on hydrophobic Y-type zeolite at various temperatures.

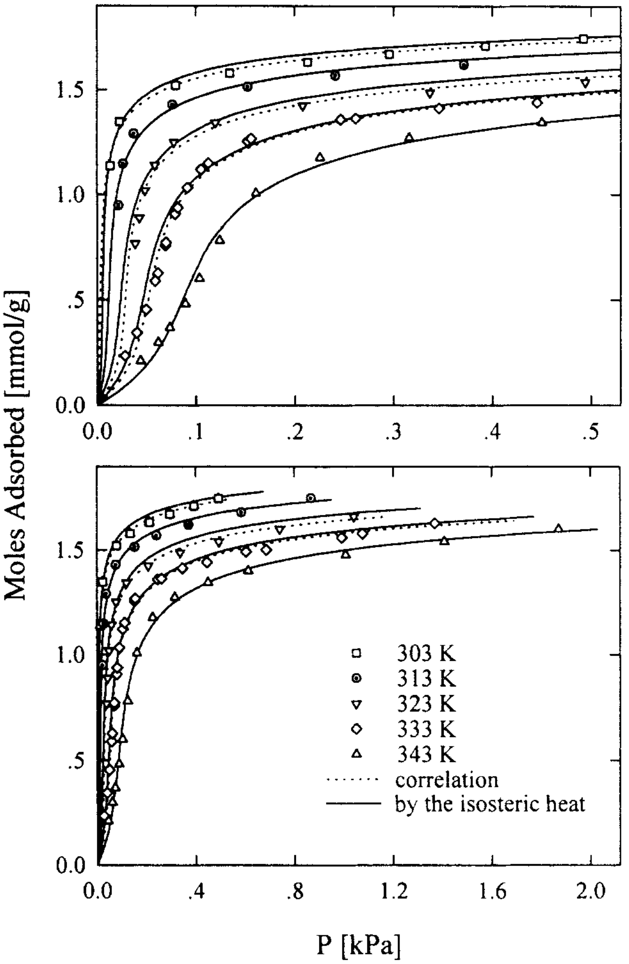

Figure 4. Adsorption isotherms for toluene on hydrophobic Y-type zeolite at various temperatures.

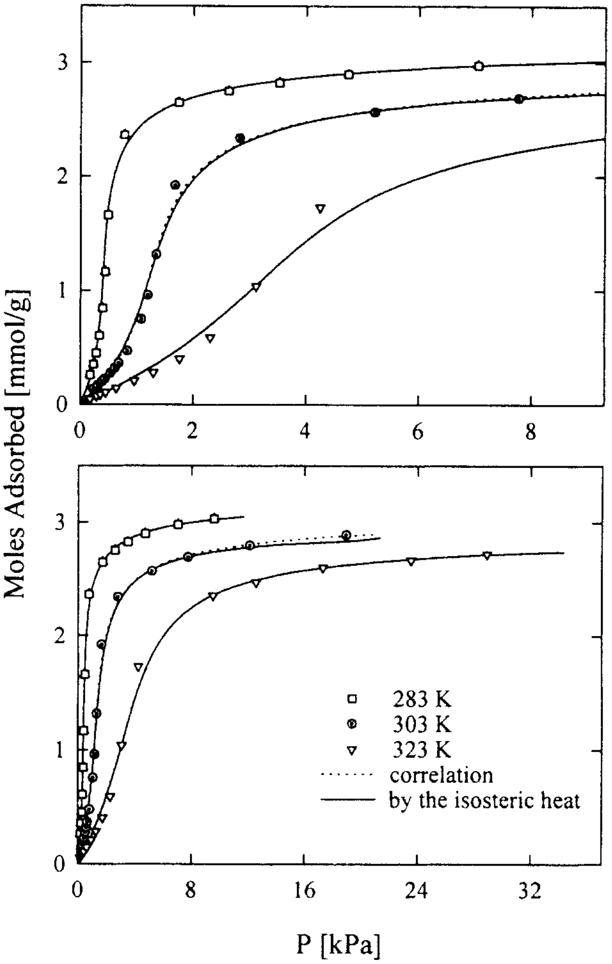

Figure 5. Adsorption isotherms for dichloromethane on hydrophobic Y-type zeolite at various temperatures.

$$q^{I}_{st}(T_1,T_2) = q^{II}_{st}(T,T_2), \tag{9}$$

where T is a specific temperature of interest and Eq. 9 can be represented as:

$$\frac{\ln P_1 - \ln P_2}{1/T_2 - 1/T_1} = \frac{\ln P - \ln P_2}{1/T_2 - 1/T} \quad (\text{constant } N), \tag{10}$$

where P is the equilibrium pressure at T. Finally one gets

$$P = \exp\left[\frac{\ln P_1 - \ln P_2}{1/T_2 - 1/T_1}\left(\frac{1}{T_2} - \frac{1}{T}\right) + \ln P_2\right] \quad (\text{constant } N). \tag{11}$$

Equation 11 enables one to estimate the adsorption equilibrium relation of P and N at T within the experimental temperature range T_1–T_2. In applying Eq. 11, two sets of isotherm equations of $P_1 = f(N)_{T_1}$ and $P_2 = f(N)_{T_2}$, which can be obtained by fitting the experimental data at two different temperatures of T_1 and T_2 are needed.

In this section, rather than using an isotherm equation that includes the parameters as a function of temperature, we applied the proposed method (Eq. 11) to predict the temperature-dependent adsorption equilibrium relation. We used our own experimental data to test benzene, toluene, dichloromethane, and 1,1-dichloro-1-fluoroethane on hydrophobic Y-type zeolite at various temperatures.

For a benzene–hydrophobic Y-type zeolite system, the equilibrium data were obtained at the temperature range of 288–373 K. With two sets of data at 288 K (T_1) and 373 K (T_2), we used Eq. 11 to calculate the equilibrium relations at the following temperatures: 303, 313, 323, 333, 343, and 358 K. The results are shown in Figure 3, with symbols representing the data, dashed lines representing the results by correlation, and solid lines representing the results by the isosteric heat of adsorption. As shown in this figure, the results by the isosteric heat of adsorption were found to be in excellent agreement with the experimental data. Even in the low pressure region, Eq. 11 gives nearly identical results compared with those of the general adsorption isotherm, which has five parameters.

For the case of toluene–hydrophobic Y-type zeolite system, the equilibrium data were obtained at temperatures of

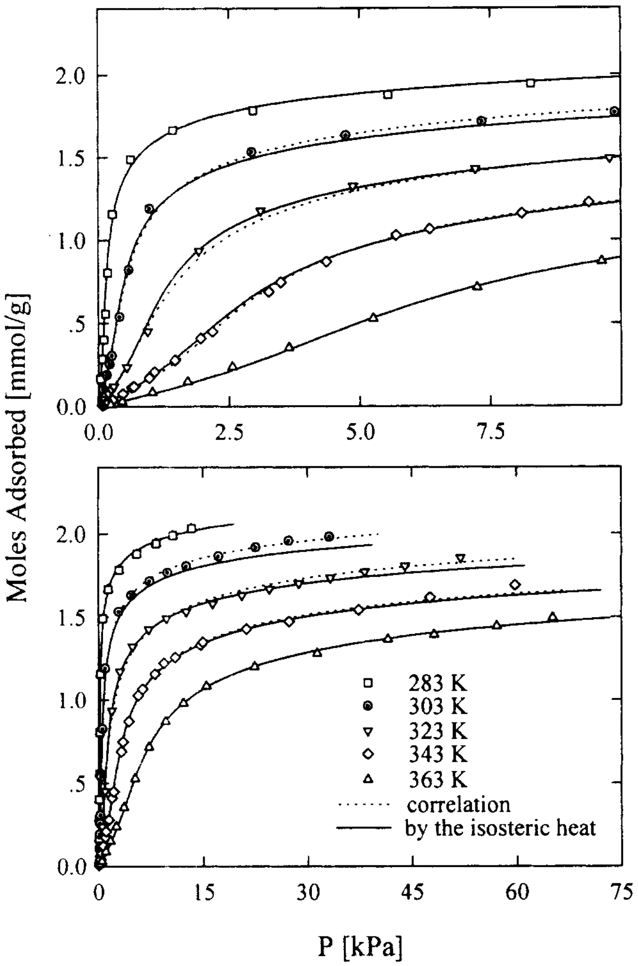

Figure 6. Adsorption isotherms for 1,1-dichloro-1-fluoroethane on hydrophobic Y-type zeolite at various temperatures.

303, 313, 323, 333, and 343 K. With two sets of data at 313 K (T_1) and 343 K (T_2), we used Eq. 11 to predict the experimental data at 303, 323, and 333 K. These results are shown in Figure 4, which demonstrates that using Eq. 11 gives a moderate prediction of the experimental data.

Our next tests used the isotherm data of dichloromethane at 283, 303, and 323 K. For the two data sets at 283 K (T_1) and 323 K (T_2), the experimental data at 303 K was predicted and we still achieved an excellent fit between the experimental data and the proposed method (Figure 5).

Finally, we tested the proposed method with the experimental data of 1,1-dichloro-1-fluoroethane on hydrophobic Y-type zeolite. The data were collected at temperatures of 283, 303, 323, 343, and 363 K. With two sets of data at 283 K (T_1) and 363 K (T_2), calculations from the proposed method were obtained at 303, 323, and 343 K, respectively. As shown in Figure 6, with the exception of the data points of 303 and 323 K in the high pressure region, the predictions of Eq. 11 are excellent.

From the preceding results, it can be stated that (1) even when the adsorption equilibrium data exhibit an unusual isotherm shape, the prediction based on isosteric heat of adsorption gives reasonable and fairly accurate results; and (2) this approach allows reasonable extrapolation outside the experimental temperature range, if the temperature is not too far outside the range.

Conclusions

This article presents the new experimental results for adsorption equilibria of some organic solvents (benzene, toluene, dichloromethane, and 1,1-dichloro-1-fluoroethane) on a hydrophobic Y-type zeolite as an alternative adsorbent for solvent recovery. The experimental isotherms were the S-shaped isotherms, which can be classified as type V. In order to predict the experimental isotherms at various temperatures simultaneously, we applied a simple thermodynamic method that is based on the theory that the isosteric heat of adsorption is independent of temperature. This method showed that the predicted value is in good agreement with the experimental data in the temperature range for the system tested. This method is capable of dealing with various adsorption systems, even when the equilibrium relationship exhibits unusual isotherm shape. Moreover, it is an easy method to apply in dynamic studies.

Notation

k = isotherm parameter of the general adsorption isotherm
M_0 = isotherm parameter of the general adsorption isotherm, $\text{mmol}\cdot\text{g}^{-1}$
N^{∞} = isotherm parameter of the general adsorption isotherm, $\text{mmol}\cdot\text{g}^{-1}$
R = gas constant, $\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$
W = isotherm parameter of the general adsorption isotherm
θ = surface coverage

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