Adsorption Equilibria of Xylene Isomers on a KBa–Y Zeolite

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Experimental data are reported for adsorption from binary and ternary liquid mixtures *p*-xylene/*m*-xylene, *p*-xylene/ethylbenzene, ethylbenzene/*m*-xylene, and *p*-xylene/*m*-xylene/ethylbenzene on a KBa–Y zeolite at 25 °C. In calculations of the difference of free energy of immersion for pure adsorbates in binary systems, three binary data have been found to be thermodynamically consistent. Based on the method of Price and Danner, predictions of adsorption equilibria for the ternary system *p*-xylene/*m*-xylene/ethylbenzene are also carried out by using only the parameters determined from the constituent binary pairs. Compared with the experimental data, the absolute average percent deviations of the predicted values of the surface excess are 6 and 6.3% for *p*-xylene and *m*-xylene, respectively. As a result, the correlations can be used to calculate the adsorption equilibrium for binary and ternary systems involving *p*-xylene, *m*-xylene, and ethylbenzene on a KBa–Y zeolite at 25 °C.

Introduction

Adsorption separation processes have been widely used in industry, particularly in the petroleum refining and petrochemical industries. Due to the nearly equal boiling points (*p*-xylene, 138.4 °C; *m*-xylene, 139.1 °C; ethylbenzene, 136.2 °C), the xylene isomers cannot be separated by distillation. The separation of the xylene isomers has become one of the most important applications of adsorption separation processes. The Parex process (*1*), for instance, has been commercialized for recovery of *p*-xylene from mixed C_8 aromatics. For recovery of ethylbenzene from mixed aromatics, the Ebex process (*1*) has been developed and demonstrated in pilot scale operation. There has been a considerable economic stimulus to develop a more efficient adsorption separation process for these isomers.

Efficient design of adsorption separators is contingent upon accurate predictions of the equilibrium mole fractions of adsorbates in solution and the adsorption separation factors among adsorbates. Typically, predictions are made by use of correlations developed from the experimental adsorption equilibrium data. Within the framework of thermodynamic models for adsorption equilibria, predictions of the multicomponent adsorption equilibria over the entire concentration range can be made by using only parameters determined from adsorption data for the constituent binary systems. Consequently, measurements of the adsorption equilibria of binary systems are of great importance and can be served as the data base from which correlations can be developed. When adsorption equilibrium data for the multicomponent systems, e.g., ternary systems or higher order systems, are available, the predictability of the correlation for multicomponent adsorption equilibria can further be justified.

The thermodynamic treatment of adsorption equilibrium from multicomponent liquid solutions on solids has progressed considerably in recent years (*2*–*12*). The method proposed by Minka and Myers (*11*) is one of the successful approaches with predictive capability. In their theory, the adsorbent is assumed to be energetically homogeneous and both the adsorbed and bulk phases are treated nonideally. For predicting adsorption equilibria for multicomponent systems, the method of Minka and Myers requires both numerical differentiation and numerical integration, which make the calculations cumbersome. Combining the thermodynamic and kinetic treatments of liquid adsorption, Price and Danner (*12*) have proposed an alternative method for predicting the multicomponent adsorption equilibria. For predictions of adsorption equilibria for the benzene/ethyl acetate/cyclohexane system on activated carbon at 303 K (*12*), the method of Price and Danner yields similar results as those obtained by the method of Minka and Myers. The calculations involved in the method of Price and Danner are, however, simpler than those of the method of Minka and Myers.

In this study, adsorption equilibria from binary and ternary liquid mixtures, *p*-xylene/*m*-xylene, *p*-xylene/ethylbenzene, ethylbenzene/*m*-xylene, and *p*-xylene/*m*-xylene/ethylbenzene on a KBa–Y zeolite at 25 °C were measured. The thermodynamic treatment of Minka and Myers is applied to analyze our adsorption equilibrium data for binary systems. The characterized activity coefficients of species in the adsorbed phase in the binary systems are evaluated. Constants of the Redlich–Kister equation (*13*) for the activity coefficients as a function of the adsorbed-phase mole fraction are determined. The adsorption equilibrium constant, as proposed by Price and Danner, are also determined for each binary system. With use of the method of Price and Danner, predictions of the adsorption equilibrium from the ternary system *p*-xylene/*m*-xylene/ethylbenzene using the parameters determined from the binary adsorption data are made and compared with the experimental data.

Experimental Section

Normally, a 5-g amount of fixed-composition binary or ternary liquid solution (xylene isomers), containing a carefully weighted 2-g amount of zeolite, is used for the adsorption equilibrium measurement. All chemicals are Riedel-deHaen reagent grade with a purity of 99% or higher. The zeolite employed in the adsorption is a KBa–Y type of 20/30 mesh. Initially, the zeolite sample is completely dried by heating at 350 °C for 8 h in a furnace equipped with a vacuum pump for eliminating the moisture contained in the zeolite. When the zeolite sample is cooled, the vacuum pump is removed, and the liquid solution is then added. Direct contact of the adsorbent with air is avoided in the experiment (*14*). The reactor is placed in a constant-temperature water bath (±0.1 °C) equipped with an oscillator. The temperature is controlled at 25 °C. The concentrations of the liquid solution are measured before and after adsorption equilibrium is reached. The concentration of the liquid sample is analyzed by a Shimadzu Model GC-4C gas chromatograph equipped with a flame ionization detector (FID). The analytical column is packed with 1.5% Bentone 34 and 1.5% didecyl phthalate on Chromosorb W 80/100 mesh. The column temperature for analyzing is set at 85 °C, and carrier gas is nitrogen with a flow rate of 40 mL/min (*15*). The surface excess of species *i* is calculated from the initial mole fraction of component *i*, the equilibrium mole fraction of component *i*, and the initial total number of moles in the bulk phase.

Calculation of the Adsorbed-Phase Activity Coefficients

The free energy of immersion is defined as the change in Gibbs free energy associated with the contact of an adsorbent

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Table I. Comparison of Experimental and Calculated Values of Surface Excess for Binary Systems

p-xylene (1)/m-xylene (2)			p-xylene (1)/ethylbenzene (2)			ethylbenzene (1)/m-xylene (2)		
x_1	$n^e_{1,exp}$, mmol/g	$n^e_{1,cal}$, mmol/g	x_1	$n^e_{1,exp}$, mmol/g	$n^e_{1,cal}$, mmol/g	x_1	$n^e_{1,exp}$, mmol/g	$n^e_{1,cal}$, mmol/g
0.025	0.361	0.370	0.038	0.103	0.103	0.025	0.171	0.201
0.035	0.512	0.521	0.075	0.172	0.174	0.050	0.331	0.319
0.046	0.645	0.649	0.108	0.205	0.233	0.082	0.435	0.451
0.062	0.775	0.763	0.137	0.282	0.289	0.145	0.595	0.592
0.101	0.851	0.906	0.185	0.325	0.350	0.175	0.635	0.626
0.160	0.905	0.963	0.215	0.389	0.385	0.225	0.695	0.648
0.265	0.928	0.905	0.278	0.425	0.423	0.283	0.671	0.640
0.370	0.825	0.796	0.390	0.423	0.419	0.375	0.576	0.573
0.475	0.681	0.673	0.475	0.392	0.391	0.472	0.435	0.478
0.579	0.541	0.544	0.582	0.325	0.332	0.585	0.335	0.388
0.686	0.402	0.408	0.661	0.262	0.266	0.691	0.242	0.285
0.792	0.263	0.271	0.768	0.158	0.185	0.781	0.182	0.205
0.892	0.125	0.136	0.885	0.068	0.097	0.890	0.102	0.115
AAD,[a] %		3.1			6.9			8.2

[a] Absolute average deviation (AAD) = $(1/N)\sum_i^N[|n^e_{1,exp} - n^e_{1,cal}|/n^e_{1,exp}] \times 100$, where N is the total number of data points.

with a liquid. For a binary system, the free energy of immersion, ϕ, can be expressed (*11*) as

$$\frac{\phi - \phi^\circ_1}{RT} = -\int_{x_1=1}^{x_1} \frac{n^e_1}{x_1 x_2 \gamma_1}\, d(\gamma_1 x_1) \qquad (1)$$

where ϕ°_1 is the free energy of immersion of the adsorbent in the pure liquid 1 and γ_1 is the activity coefficient in the bulk liquid phase. The surface excess of component 1, n^e_1, is defined (*16*) as

$$n^e_1 = n^\circ(x^\circ_1 - x_1) \qquad (2)$$

where n° is the initial total number of moles in the bulk phase, x°_1 is the initial mole fraction of component 1, and x_1 is the equilibrium mole fraction of component 1. From eq 2, the surface excess of the components can be calculated from measurable quantities, n°, x_1, and x°_1.

Providing that the bulk-phase activity coefficients are available, the free energy of immersion of the adsorbent in solution can be calculated from eq 1. When the free energies of immersion are available, the adsorbed-phase activity coefficients can then be obtained as follows:

$$\gamma'_1 = \frac{x_1\gamma_1}{x'_1} \exp\left[\frac{\phi - \phi^\circ_1}{m_1 RT}\right] \qquad (3)$$

$$\gamma'_2 = \frac{x_2\gamma_2}{x'_2} \exp\left[\frac{\phi - \phi^\circ_2}{m_2 RT}\right] \qquad (4)$$

where x'_i is the mole fraction of component i in the adsorbed phase and the prime denotes the adsorbed phase. The obtained adsorbed-phase activity coefficients are then fitted by using the four-constant Redlich–Kister equation.

Thermodynamic Consistency Test

The difference in free energies of immersion provides a useful test of thermodynamic consistency for binary adsorption isotherms (*17*). If adsorption data for three binaries, (1 + 2), (2 + 3), and (3 + 1), are all measured, the test of consistency is

$$(\phi^\circ_2 - \phi^\circ_1) + (\phi^\circ_3 - \phi^\circ_2) + (\phi^\circ_1 - \phi^\circ_3) = 0 \qquad (5)$$

The calculated values of the left-hand side of eq 5 can be used as a guideline for a consistency test for binary adsorption data.

Prediction of Adsorption Equilibria for Ternary Systems

The thermodynamic treatment of Minka and Myers (*11*), for the adsorption from binary mixtures, can be extended directly to ternary systems. The extension is straightforward. The characterized activity coefficients of species in the adsorbed phase, determined from each binary pair, are required in the calculation; there is no need to determine parameters from ternary systems. However, the disadvantage of the extension to multicomponent liquid adsorption equilibria is the complexity of the calculation procedure in which both numerical differentiation and integration are involved. Thus, for multicomponent liquid adsorption equilibria predictions, we adopted the method proposed by Price and Danner (*12*). The method of Price and Danner combines the thermodynamic and kinetic treatments of liquid adsorption. Compared to the method of Minka and Myers, the calculation procedure is simpler.

Price and Danner proposed an equation for the exchange of molecules between the adsorbed and bulk liquid phases as follows:

$$r_{ji}(i) + (j)' \rightleftharpoons r_{ji}(i)' + (j) \qquad (6)$$

where (i) and (j) denote the ith and jth components, respectively; the prime represents the adsorbed phase. The r_{ji} is the ratio of the adsorbent capacity of pure adsorbates j and i, and can be calculated as

$$r_{ji} = m_i/m_j \qquad (7)$$

For adsorption systems involving microporous adsorbent with homogeneous adsorbed phase, the adsorption equilibrium constant can be expressed (*12*) as

$$K_{ij} = [(\gamma_j\theta_j)/(\gamma_i\theta_i)^{r_{ij}}][(\gamma'_i\theta'_i)^{r_{ij}}/(\gamma'_j\theta'_j)] \qquad (8)$$

where θ_i and θ'_i are the liquid-phase volume fractions and adsorbed-phase volume fractions, respectively. An optimal adsorption equilibrium constant K_{12} is determined for each binary system by minimizing the sum of the squared differences between the experimental and correlated surface excess values (*12*). For predictions of the surface excess for ternary systems the parameters required are constants of the Redlich–Kister equation and the adsorption equilibrium constant for each binary system. The calculation procedure for predicting surface excess values in a ternary system for a given bulk liquid-phase composition is available elsewhere (*12*).

Results and Discussion

Experimental data for adsorption from three binary liquid xylenes p-xylene/m-xylene, p-xylene/ethylbenzene, and ethylbenzene/m-xylene on a KBa-Y zeolite at 25 °C are presented in Table I. Plots of the measured surface excess versus the liquid composition are also shown in Figures 1–3. The data of the adsorption equilibrium from the ternary system

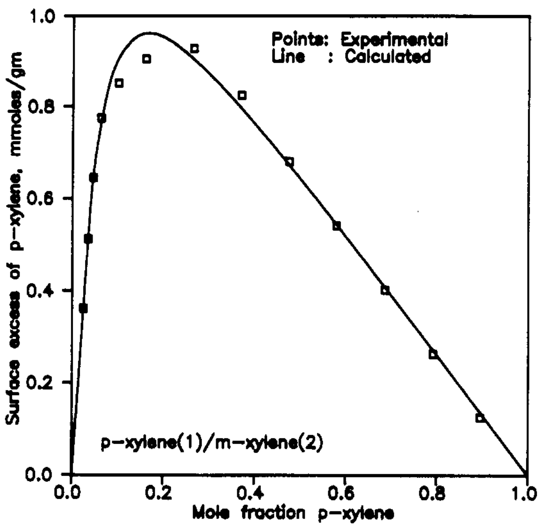

Figure 1. Surface excess of *p*-xylene adsorbed on a KBa-Y zeolite from a liquid mixture of *p*-xylene (1)/*m*-xylene (2) at 25 °C. The solid line is calculated by the method of Minka and Myers (*11*).

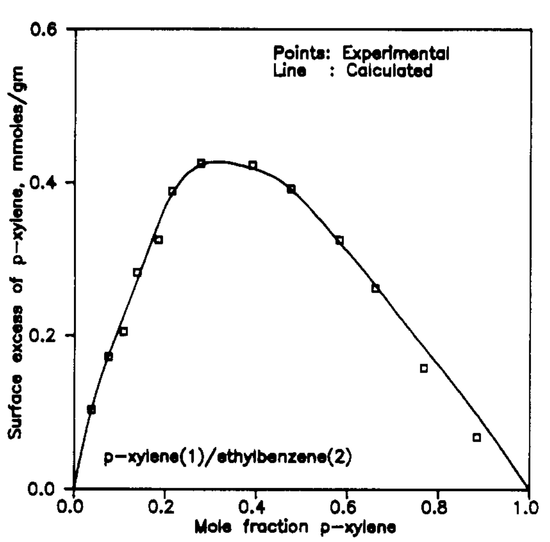

Figure 2. Surface excess of *p*-xylene adsorbed on a KBa-Y zeolite from the liquid mixture of *p*-xylene (1)/ethylbenzene (2) at 25 °C. The solid line is calculated by the method of Minka and Myers (*11*).

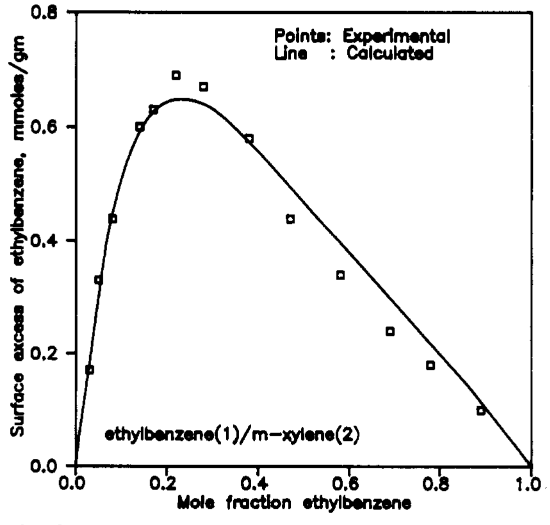

Figure 3. Surface excess of ethylbenzene adsorbed on a KBa-Y zeolite from the liquid mixture of ethylbenzene (1)/*m*-xylene (2) at 25 °C. The solid line is calculated by the method of Minka and Myers (*11*).

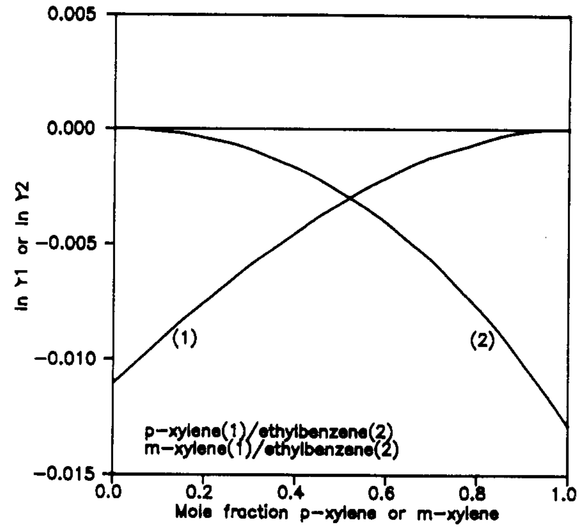

Figure 4. Activity coefficients, calculated by using UNIFAC (*18*), in bulk liquid phase for binary mixtures at 25 °C.

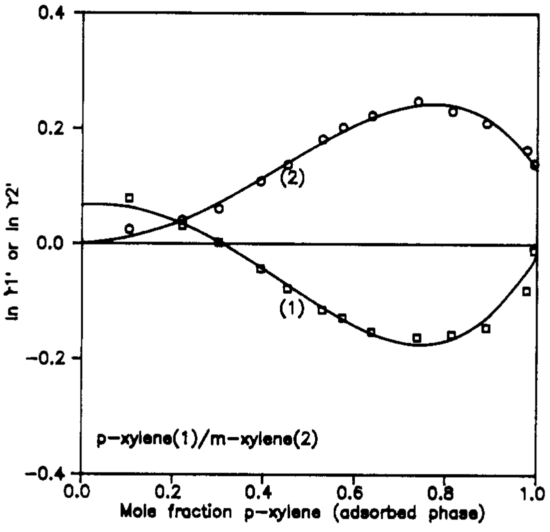

Figure 5. Adsorbed-phase activity coefficients for the system *p*-xylene (1)/*m*-xylene (2). Points: experimental values. Solid lines: smoothed values.

Table II. Capacity of KBa-Y Zeolite at 25 °C

adsorbate	m_i,[a] mmol/g
p-xylene	1.32
m-xylene	0.84
ethylbenzene	1.13

[a] Reference 14.

p-xylene/*m*-xylene/ethylbenzene on a K-BaY zeolite at 25 °C are reported in Table VI.

In this study, the activity coefficients of components in the bulk liquid phase, required in the calculations, are calculated by using the UNIFAC (*18*) method, a group contribution method for estimating the activity coefficients. In Figure 4, the activity coefficients calculated from the UNIFAC method are shown. Both the *p*-xylene/ethylbenzene and the *m*-xylene/ethylbenzene systems show negative deviations from Raoult's law in the bulk liquid phase. Since *p*-xylene and *m*-xylene are isomers, the UNIFAC gives the ideal solution behavior, i.e., the activity coefficients are equal to unity. The molar capacity of the KBa-Y zeolite for pure components, required in the calculation, are given in Table II (*14*).

For a particular binary system, the equilibrium mole fraction of species in the adsorbed phase can be calculated from the

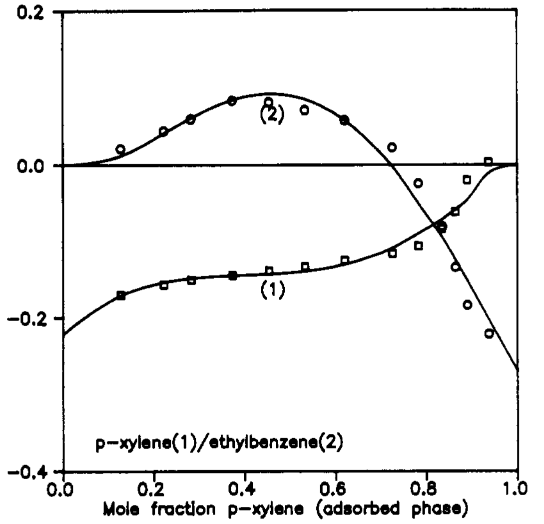

Figure 6. Adsorbed phase activity coefficients for the system *p*-xylene(1)/ethylbenzene(2). Points: experimental values. Solid lines: smoothed values.

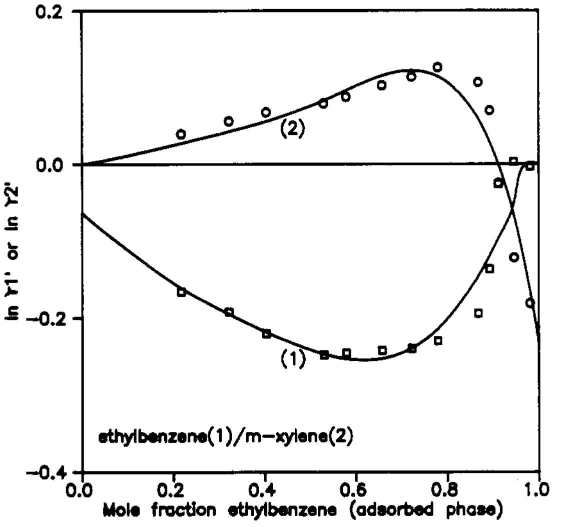

Figure 7. Adsorbed-phase activity coefficients for the system ethylbenzene (1)/*m*-xylene (2). Points: experimental values. Solid lines: smoothed values.

Table III. Constants of the Redlich–Kister Equation for Binary Systems in the Adsorbed Phase at 25 °C

constant	*p*-xylene (1)/ *m*-xylene (2)	*p*-xylene (1)/ ethylbenzene (2)	ethylbenzene (1)/ *m*-xylene (2)
A	0.1303	−0.1317	−0.3114
B	−0.4233	−0.7512	−0.6984
C	−0.7869	−0.0974	−0.4776
D	−0.6812	0.9605	−0.2151

Table IV. Thermodynamic Consistency Test of Binary Systems at 25 °C

binary systems	differences in free energy of immersion, cal/g
p-xylene (1)/*m*-xylene (2)	$(\phi^\circ_1 - \phi^\circ_2)/RT = -3.997$
ethylbenzene (3)/*p*-xylene (1)	$(\phi^\circ_3 - \phi^\circ_1)/RT = +1.523$
m-xylene (2)/ethylbenzene (3)	$(\phi^\circ_2 - \phi^\circ_3)/RT = +2.486$
	sum = +0.012

Table V. Optimal Adsorption Equilibrium Constants for Binary Systems

liquid mixture	K_{21}	n_1^s AAD, %
p-xylene (1)/*m*-xylene (2)	0.0471	4.8
p-xylene (1)/ethylbenzene (2)	0.2569	9.0
ethylbenzene (1)/*m*-xylene (2)	0.1124	11.0

Table VI. Comparison of Experimental and Predicted Values of Surface Excess for the *p*-Xylene (1)/*m*-Xylene (2)/Ethylbenzene (3) System on a KBa–Y Zeolite at 25 °C

liquid mole fraction		surface excess, mmol/g			
		p-xylene		*m*-xylene	
x_1	x_2	exp	calc	exp	calc
0.0921	0.8153	0.7054	0.6912	−0.8020	−0.8142
0.1222	0.0944	0.4840	0.4335	−0.1046	−0.1047
0.1945	0.6750	0.7699	0.6964	−0.6458	−0.7533
0.2447	0.1251	0.3489	0.3517	−0.1224	−0.1449
0.3889	0.2031	0.0262	0.0244	−0.2263	−0.2336
0.5026	0.4142	0.5207	0.5162	−0.4561	−0.4951
0.6005	0.2489	0.3841	0.3586	−0.2692	−0.3041
0.6817	0.1848	0.3323	0.2882	−0.2100	−0.2271
0.8045	0.1001	0.2026	0.1946	−0.1212	−0.1239
AAD,[a] %			6.0		8.3

[a] AAD = absolute average deviation.

experimental surface excess, the mole fraction in the liquid phase and the adsorbent capacity of pure species (*11*). The free energy of immersion in the mixture can be calculated by numerical integration of the right-hand side of eq 1. From eqs 3 and 4, the activity coefficients of species in the adsorbed phase can then be evaluated. The calculated activity coefficients in the adsorbed phase for three binaries are shown in Figures 5–7. The activity coefficients in the adsorbed phase show complicated variation with the mole fraction of the species in the adsorbed phase. The constants of the Redlich–Kister equation are determined from the activity coefficients of species in the adsorbed phase and are presented in Table III.

From the calculated activity coefficients of species in the adsorbed phase, the surface excess of species *i* can be calculated by using the iterative procedure (*11*). In Table I, the calculated surface excess for three binaries are presented. The calculated surface excess are also shown graphically in Figures 1–3. The average absolute percentage deviations for the surface excess of the first component are 3.1, 6.9, and 8.2% for *p*-xylene/*m*-xylene, *p*-xylene/ethylbenzene, and ethylbenzene/*m*-xylene, respectively. The results show that the adsorption equilibrium data of xylene binaries can be well correlated by the method of Minka and Myers.

The difference of free energy of immersion of the adsorbent between two pure liquids are calculated and presented in Table IV. The summation of the three binaries is equal to 0.012, which is less than 1% of the value of the largest difference in free energies. This indicates that the data exhibit a high degree of thermodynamic consistency.

For predictions of the adsorption equilibrium for ternary systems, the method of Price and Danner is applied. The determined adsorption equilibrium constants for each binary system are presented in Table V. The average absolute percentage deviations for the calculated surface excess of the first component are 4.8, 9, and 11% for *p*-xylene/*m*-xylene, *p*-xylene/ethylbenzene, and ethylbenzene/*m*-xylene system, respectively. The results are in the same order as those obtained by the method of Minka and Myers.

With the characterization adsorbed-phase activity coefficients and the adsorption equilibrium constants, both determined from the adsorption equilibrium data of binary systems, the method of Price and Danner can be used directly to predict the adsorption equilibria for ternary systems. The results of the predicted surface excess of the ternary system *p*-xylene/*m*-xylene/ethylbenzene are presented in Table VI. The average absolute percentage deviations for the surface excess for the *p*-xylene and *m*-xylene are 6.0 and 8.3%, respectively. The predictions are quite satisfactory. When the mole fraction ratios of *m*-xylene/ethylbenzene in the liquid phase are fixed at 0, 0.5, 1, 2, 3, and ∞, the predicted values of surface excess of *p*-

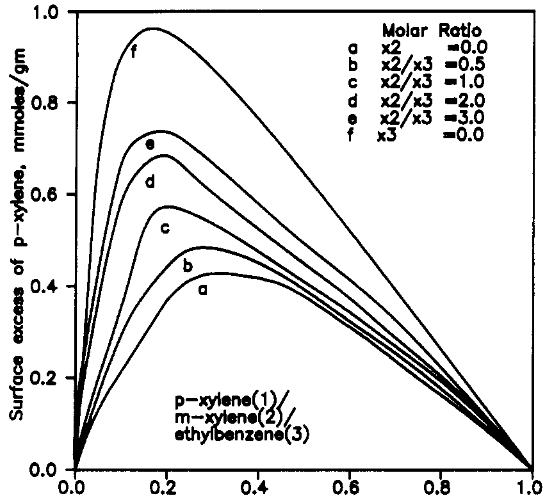

Figure 8. Predictions of the surface excess of *p*-xylene adsorbed on a KBa-Y zeolite at 25 °C using the method of Price and Danner (*12*) for the ternary system *p*-xylene (1)/*m*-xylene (2)/ethylbenzene (3) at various bulk-phase mole fraction ratios: $x_2/x_3 = 0, 0.5, 1, 2, 3$, and ∞.

xylene adsorbed for the *p*-xylene/*m*-xylene/ethylbenzene system are shown graphically in Figure 8. The calculated surface excess for *p*-xylene shows a systematic trend from the *p*-xylene/*m*-xylene, where the mole fraction of ethylbenzene is equal to zero, to the system *p*-xylene/ethylbenzene, where the mole fraction of *m*-xylene is equal to zero.

Conclusions

Adsorption equilibria data from binary and ternary liquid mixtures *p*-xylene/*m*-xylene, *p*-xylene/ethylbenzene, ethylbenzene/*m*-xylene, and *p*-xylene/*m*-xylene/ethylbenzene on a K-BaY zeolite at 25 °C are presented. The thermodynamic models for describing adsorption equilibrium have been applied to analyze the data. With calculation of the free energy of immersion of the adsorbent for two pure adsorbates, the three binary adsorption equilibrium data have been found to be thermodynamically consistent. For each binary system, the characterized activity coefficients in the adsorbed phase and the adsorption equilibrium constant have been determined. Calculation of adsorption equilibrium for binary systems yields satisfactory results. Predictions of the adsorption equilibrium for the ternary system *p*-xylene/*m*-xylene/ethylbenzene are also carried out based on the method of Price and Danner. There is no need to determine parameters from ternary systems. Compared with the experimental data, the results are quite satisfactory. This demonstrates that the adsorption equilibrium data are well correlated by the thermodynamic models studied in this work. Consequently, the parameters obtained in this work, within the framework of the thermodynamic model for adsorption equilibrium, can be used to perform calculations of adsorption equilibrium from binary and ternary systems consisting of *p*-xylene, *m*-xylene, and ethylbenzene on a KBa-Y zeolite at 25 °C.

Nomenclature

K_{ij}	optimal adsorption equilibrium constant
m_i	capacity of adsorbent for pure ith liquid, mmol/g
n	number of moles per unit mass of adsorbent, mmol/g
n_i^e	surface excess of component i, mmol/g
r_{ji}	ratio of adsorbent capacities of pure j and i components
R	gas constant, 8.314 J/(mol K)
T	absolute temperature, K
x_i	mole fraction of species i in mixture

Greek Symbols

θ_i	volume fraction of component i
γ_i	activity coefficient of component i
ϕ	free energy of immersion of adsorbent in mixture, J/kg

Superscripts

$^\circ$	refers to pure adsorbate, refers to adsorbate solution before contacting adsorbent
$'$	refers to adsorbed phase

Subscripts

calc	calculated
exp	experimental
i	refers to component i

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