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Vapor Phase Adsorption Equilibria of Toluene + 1-Propanol Mixtures on Y-Zeolites with Different Silicon to Aluminum Ratios

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Zeolites with various silicon to aluminum ratios usually exhibit polar and nonpolar adsorption behavior depending on the degree of dealumination. The objective of the present study was to show the influence of the surface polarity on the adsorbate phase composition containing a polar and a nonpolar component: toluene and 1-propanol, respectively. Such systematic investigations should help to understand the significance of some adsorbent parameters, such as the surface heterogeneity. This basic knowledge is important for the development of improved models for the prediction of multicomponent adsorption equilibria.

Introduction

Highly dealuminated Y-zeolites—here abbreviated as DAY—have been successfully tested in adsorption pilot plants as a good alternative to the conventionally used activated carbon (*1*). The main areas of application for this new type of adsorbent are adsorptive waste gas purification and organic solvent recovery.

The design of any adsorption process requires the knowledge of adsorption equilibria and the kinetics of mass transfer in the adsorbent pellet. Recently, it has been shown that it is easy to predict adsorption and desorption breakthrough behavior of a nonisothermal fixed bed adsorber using only adsorption equilibrium data and tortuosity factors obtained from single-pellet experiments (*2*).

In most cases industrial waste gas streams contain more than one adsorbable component. Therefore, the knowledge of multicomponent equilibria is of great interest. It is time consuming and expensive to measure adsorption equilibria of binary and higher systems, and up to now it was not possible to predict them with the required accuracy using only pure component adsorption equilibrium data. Among others, the difficulties for prediction are based on the lack of information about the influence of the surface properties on adsorption equilibria.

The aim of the present work is to show to what extent the surface hydrophobicity of Y-zeolites, e.g., the silicon to aluminum ratio, can influence the composition of a binary mixture containing a polar and a nonpolar component. The systematic investigations were performed to clarify the ability of the ideal adsorbed solution theory (IAST) from Myers and Prausnitz (*3*) for predicting the binary adsorption data under these varying conditions. From a comparison between IAST predictions and correlation results using the real adsorbed solution theory (RAST) (*4*), it should be demonstrated that the information of surface hydrophobicity is almost completely taken into account by the pure adsorption isotherms.

Experimental Section

Materials. Samples of dealuminated Y-zeolites (DAY) with three different silicon to aluminum ratios, 13 (designated as DAY-13), 55 (designated as DAY-55), and 100 (designated as DAY-100), were supplied by Degussa AG, Hanau, FRG. At Degussa AG these zeolite samples were progressively dealuminated using the $SiCl_4$ vapor method of Beyer and Belenykaja (*5*). The zeolite powder was pressed without any binder, crushed, and sieved. The collected particle size fraction ranged between 0.20 and 0.315 mm.

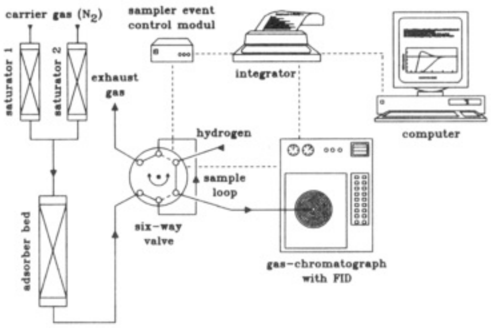

Figure 1. Scheme diagram of the flow-type apparatus for vapor adsorption experiments.

For the adsorption experiments toluene (ACS certified 99.7%) and 1-propanol (99.5% GC purity) were used as adsorbates. Both chemicals were supplied by Aldrich Chemical Co. Dry nitrogen was used as the carrier gas with a purity greater than 99.999%.

Apparatus. A flow-type apparatus, based upon a simple chromatographic technique with frontal analysis, was used for measuring both single- and mixed-vapor adsorption equilibria. A schematic diagram of the experimental arrangement is shown in Figure 1.

All experiments were performed at atmospheric pressure. The gaseous feed mixture was generated by passing a carrier gas (nitrogen) through two parallel saturators filled with toluene and 1-propanol, respectively. The saturators were thermostated to the required low temperature using a cryostat to obtain the desired partial pressures of the adsorptives in the gas stream. All gas lines and valves were heated to avoid condensation of the vapors. After mixing, the gas stream passed a glass-made adsorption chamber thermostated at 298.15 and 318.15 K. Isothermal conditions were controlled with Pt-100 thermocouples. In all experiments the mass of the dry adsorbent was $m_{\text{ads}} = 2.0$ g. The total gas flow measured at ambient temperature was about $\dot{V} = 1.0$ cm^3/s.

After the gas stream passed the adsorber bed, the concentrations of the adsorptives in the outlet gas stream

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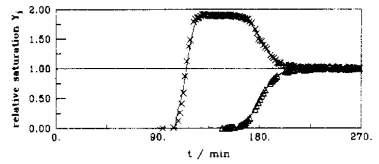

Figure 2. Typical breakthrough curves of binary mixture adsorption on DAY-zeolite. (×, 1-propanol; △, toluene).

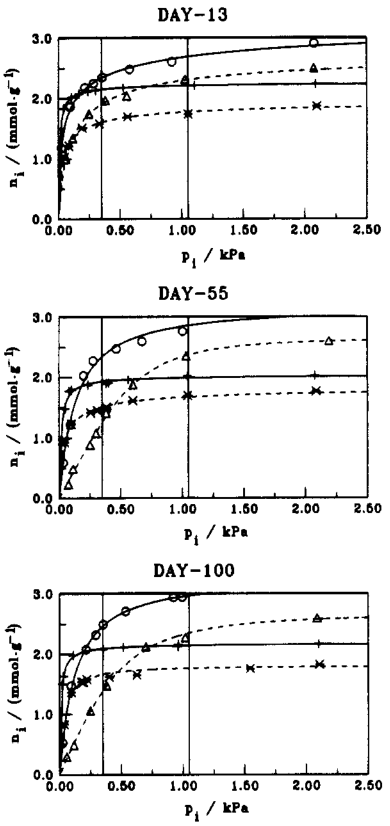

Figure 3. Moles adsorbed n_i for toluene and 1-propanol on DAY-13, DAY-55, and DAY-100 as a function of the partial pressure of adsorbate p_i (1-propanol, ○, 298.15 K; △, 318.15 K; toluene, +, 298.15 K; *, 318.15 K; —, Tóth equation 298.15 K; - - -, Tóth equation 318.15 K). Vertical lines represent constant total pressure P of binary adsorption equilibria.

were measured by using an on-line capillary gas chromatograph (column OV-1701-DF, Macherey-Nagel), which was connected with a six-way valve. The content of the six-way valve sample loop was periodically injected into the gas chromatograph and analyzed by a flame ionization detector (FID). The FID signals of both components were recorded on the integrator to obtain the desired adsorptive breakthrough curves. A typical breakthrough is shown in Figure 2. From a simple mass balance equation the total adsorbed amount and the adsorbate mole fractions could be calculated.

The adsorbent was regenerated by flushing the adsorption chamber with pure nitrogen at a temperature ofapproximately 573 K until neither adsorbate was detected by GC. After this, an increase of the desorption temperature up to 623 K ensured the completeness of the adsorbent regeneration.

The reliability and reproducibility of the experimental method were checked several times. After an adsorption experiment, the adsorbed components were collected in a cooling trap in the desorption step. A gravimetric and chromatographical analysis of the condensed samples showed deviations of less than 5% compared to the above described calculation procedure via the mass balance equation.

Table 1. Adsorption Results for the Pure Components on Dealuminated Y-Zeolites at Different Temperatures

	toluene		1-propanol	
T/K	p_i/kPa	n_i/(mmol·g^{-1})	p_i/kPa	n_i/(mmol·g^{-1})
		DAY-13		
298.15	0.0452	1.8315	0.0241	1.1809
	0.1075	2.0218	0.0953	1.8648
	0.1691	2.0786	0.2140	2.1810
	0.2391	2.1203	0.2843	2.2503
	0.3031	2.1396	0.3561	2.3545
	0.5270	2.1782	0.5778	2.4859
	1.1032	2.2142	0.9203	2.6113
	2.0885	2.2384	2.0754	2.9144
318.15	0.0457	1.0321	0.0607	1.0026
	0.0867	1.2131	0.1153	1.3419
	0.1930	1.5169	0.2475	1.7521
	0.3329	1.5848	0.3823	1.9688
	0.5623	1.7011	0.5567	2.0436
	1.0526	1.7416	1.0265	2.3164
	2.0994	1.8714	2.0753	2.5044
		DAY-55		
298.15	0.0487	1.4847	0.0328	0.5923
	0.0810	1.7692	0.0936	1.2285
	0.1025	1.7938	0.1984	2.0299
	0.2351	1.8730	0.2766	2.2752
	0.3830	1.8968	0.4652	2.4736
	0.4025	1.9074	0.6745	2.5973
	0.5627	1.9617	1.0026	2.7645
	1.0414	2.0218	2.0624	3.1280
	2.0771	2.0274		
318.15	0.0459	0.9290	0.0756	0.2318
	0.0986	1.2365	0.1126	0.4930
	0.2564	1.4195	0.2513	0.8875
	0.3093	1.4567	0.2992	1.0807
	0.3849	1.5094	0.3822	1.4163
	0.5996	1.6165	0.6021	1.8779
	1.0437	1.7002	1.0311	2.3522
	2.0876	1.7676	2.1908	2.6032
		DAY-100		
298.15	0.0227	1.5070	0.0261	0.5281
	0.0324	1.6330	0.0950	1.4752
	0.1099	1.9780	0.2144	2.0776
	0.3533	2.0762	0.2934	2.3183
	0.5164	2.1100	0.3539	2.4939
	0.9583	2.1250	0.5366	2.7115
	0.9622	2.1567	0.9231	2.9488
	2.1005	2.1668	0.9944	2.9648
			2.0882	3.1046
318.15	0.0400	0.8345	0.0584	0.2965
	0.0969	1.3692	0.1165	0.4891
	0.1785	1.5396	0.2498	1.0605
	0.1924	1.5369	0.3810	1.4780
	0.2206	1.5812	0.6995	2.1241
	0.4011	1.6221	1.0196	2.2748
	0.6250	1.6519	2.0883	2.5998
	1.5464	1.7531		
	2.1077	1.8222		
	2.9954	1.8595		

Results and Discussion

(1) Pure Component Isotherms. The adsorption isotherms for pure toluene and 1-propanol at 298.15 and 318.15 K on all three DAY samples are shown in Figure 3. The solid and dotted lines represent the best fit of the experimental adsorption data given in Table 1 using the adsorption isotherm developed by Tóth (*6*):

$$n_i = \frac{n_{mi}\, p_i}{(b_i + p_i^{c_i})^{1/c_i}} \qquad (1)$$

where n_i = amount adsorbed, n_{mi} = monolayer saturation

Table 2. Fitted Isotherm Parameters and Average Deviations

T/K	Tóth parameter	DAY-13	DAY-55	DAY-100
		Toluene		
298.15	n_{mi}/(mmol·g^{-1})	2.2651	2.0278	2.1805
318.15		1.9721	1.8405	1.8029
298.15	b_i/kPa^{c_i}	0.0174	0.0172	0.0148
318.15		0.0714	0.0694	0.0290
298.15	c_i	0.7289	0.9382	0.8502
318/15		0.6511	0.6966	1.1984
298.15	B_{is}/(J·g^{-1}·kPa^{-1})	1450.9	381.51	765.50
318.15		300.39	224.02	91.155
298.15	av dev of amt/%	0.13	1.99	0.56
318.15		1.56	1.85	2.74
		1-Propanol		
298.15	n_{mi}/(mmol·g^{-1})	3.5031	3.1853	3.2741
318.15		2.7834	2.6444	2.6646
298.15	b_i/kPa^{c_i}	0.1181	0.1266	0.1153
318.15		0.1455	0.3782	0.3242
298.15	c_i	0.4186	1.0626	1.0685
318.15		0.7082	2.6393	2.0721
298.15	B_{is}/(J·g^{-1}·kPa^{-1})	1428.9	55.206	61.304
318.15		111.92	10.105	12.142
298.15	av dev of amt/%	1.42	3.90	1.49
318.15		0.92	6.20	3.35

amount adsorbed, p_i = adsorptive partial pressure, and b_i and c_i = specific constants. The parameters of the Tóth equation were determined by fitting the experimental adsorption data using a nonlinear regression procedure based on the Simplex algorithm by Nelder and Mead (*7*).

The three-parameter equation by Tóth is often used to describe pure adsorption equilibrium data in microporous materials. Also the present investigation showed that the equation gives an excellent fit for all systems as can be seen in Table 2. Another great advantage of this equation is that it confirms the key physical constraint to be defined up to low adsorptive partial pressures. This makes it possible to calculate the second virial coefficient of adsorption B_{is} and also allows a strict thermodynamically consistent implementation in multicomponent adsorption models, like IAST or RAST (*8*):

$$B_{is} = RTn_{mi}b_i^{(-1/c_i)} \tag{2}$$

where B_{is} = second virial coefficient of component i, R = universal gas constant, and T = adsorption temperature.

The influence of the aluminum content on the adsorption behavior of toluene on all three dealuminated samples is insignificant. The adsorption data of toluene could be fitted also with any other Langmuir-type equation at least for the pressure range covered. The more hydrophilic component 1-propanol shows an evident dependence of the adsorption behavior with varying Si/Al ratios. In the Henry's law region, at low partial pressures, the slope of the isotherm increases dramatically on DAY-13 compared to DAY-55 and DAY-100 (see also the second virial coefficient given in Table 2). This behavior can be explained by an interaction of the more hydrophilic component 1-propanol with the greater electrostatic field in DAY-13 caused by the higher aluminum content (*9*). Also the saturation loading shows a similar trend. For the more hydrophilic zeolite DAY-13 the saturation loading is greater than for the more hydrophobic samples DAY-55 and DAY-100. This observation could easily be explained with a decrease of the unit cell dimension with progressive dealumination (*10*). The overcrossing of the toluene- with the 1-propanol isotherm in all cases may also indicate that there might be an adsorption azeotropic point in the binary system.

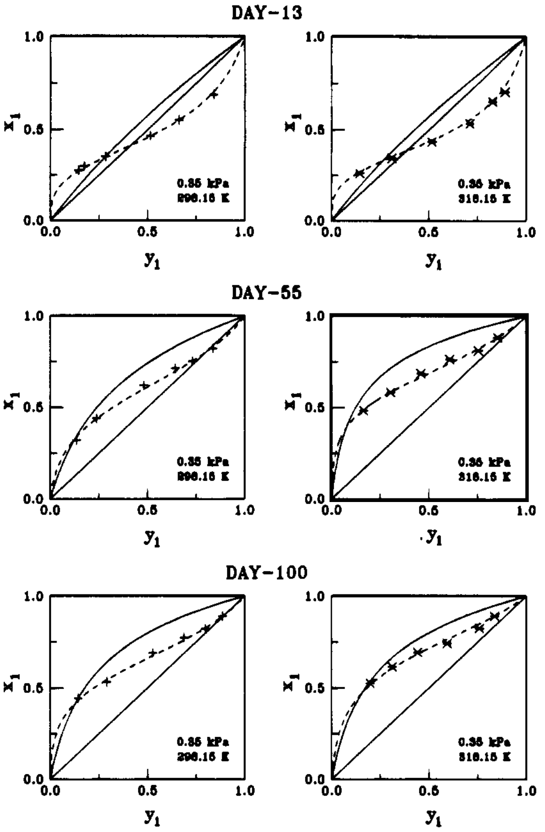

Figure 4. Binary adsorption equilibrium diagrams of toluene (1) + 1-propanol (2) on DAY-13, DAY-55, and DAY-100 at 0.35 kPa and 298.15 and 318.15 K (+, *, experimental values; —, IAST prediction; - - -, RAST correlation).

(2) Binary Mixture Isotherms. The equilibrium composition diagrams for the mixture adsorption on all three DAY samples are shown in Figures 4 and 5. Measurements were carried out at constant total pressures P (the sum of adsorptive partial pressures) of 0.35 and 1.05 kPa and temperatures of 298.15 and 318.15 K. The experimental data for the equilibrium vapor and adsorbed phase composition and the total amounts adsorbed are presented in Table 3.

To correlate the experimental data in the entire composition range, the RAS theory (*4*) and the UNIQUAC model (*11*) for the adsorbate phase activity coefficients were utilized:

$$y_iP = x_i\gamma_iP_i^0(\pi^*) \tag{3}$$

where P = total pressure (sum of the adsorptive partial pressure in the mixture adsorption), $P_i^0(\pi^*)$ = adsorptive saturation pressure of component i at the mixture spreading pressure, x_i = adsorbate phase mole fraction, y_i = vapor phase mole fraction, γ_i = activity coefficient of component i in the adsorbate phase.

The dependency of the activity coefficient γ_i on the mixture spreading pressure π^*, e.g., the expression $(\partial \ln \gamma_i/\partial\pi^*)_{x_i}$, was evaluated by the following approximation:

$$\frac{\partial \ln \gamma_i}{\partial \pi^*} = \lim_{\Delta x_i \to 0} \frac{\ln \gamma_i(x_i(\pi_i^*)) - \ln \gamma_i(x_{i-1}(\pi_{i-1}^*))}{\pi_i^* - \pi_{i-1}^*} \tag{4}$$

$$\Delta x_i = x_i(\pi_i^*) - x_{i-1}(\pi_{i-1}^*)$$

In a least squares optimization with the objective function F the interaction parameters were obtained using the

Table 3. Binary Adsorption Data for Toluene (1) + 1-Propanol (2) at Different Temperatures on Dealuminated Y-Zeolites

T/K	*P*/kPa	n_t/(mmol·g^{-1})	y_1	x_1	*T*/K	*P*/kPa	n_t/(mmol·g^{-1})	y_1	x_1
DAY 13									
298.15 K	0.3553	2.4339	0.1451	0.2755	318.15 K	0.3602	2.0457	0.1441	0.2614
	0.3573	2.4539	0.1734	0.2993		0.3641	2.0216	0.3106	0.3430
	0.3582	2.4311	0.2825	0.3520		0.3696	1.9690	0.5154	0.4352
	0.3648	2.3596	0.5130	0.4670		0.3741	1.9218	0.7107	0.5355
	0.3692	2.3448	0.6612	0.5530		0.3797	1.8461	0.8287	0.6531
	0.3746	2.2733	0.8411	0.6899		0.3801	1.7961	0.8911	0.7075
	1.0580	2.5763	0.1693	0.2697		1.0576	2.2601	0.1693	0.2501
	1.0566	2.5417	0.2832	0.3319		1.0586	2.1959	0.2873	0.3111
	1.0594	2.5058	0.4365	0.3908		1.0593	2.1789	0.4358	0.3633
	1.0647	2.4711	0.5763	0.4463		1.0631	2.1267	0.5597	0.4440
	1.0548	2.4393	0.7172	0.5396		1.0614	2.1192	0.7090	0.5321
	1.0618	2.3553	0.8305	0.6249		1.0604	2.0236	0.8460	0.6607
DAY-55									
298.15 K	0.3535	2.2467	0.1356	0.3220	318.15 K	0.3633	1.6941	0.1662	0.4832
	0.3452	2.2375	0.2368	0.4490		0.3645	1.7622	0.3044	0.5836
	0.3602	2.1845	0.4806	0.6208		0.3660	1.7286	0.4585	0.6863
	0.3616	2.1183	0.6425	0.7145		0.3674	1.6342	0.6072	0.7640
	0.3569	2.0611	0.7303	0.7514		0.3690	1.5889	0.7541	0.8120
	0.3561	2.0593	0.8375	0.8223		0.3702	1.6101	0.8535	0.8827
	1.0458	2.5655	0.1585	0.2746		1.0424	2.0755	0.1594	0.4834
	1.0427	2.3247	0.2979	0.4547		1.0457	1.9858	0.3059	0.5787
	1.0424	2.3196	0.4588	0.5565		1.0486	1.9485	0.4133	0.6153
	1.0424	2.2498	0.5961	0.6732		1.0545	1.8600	0.5951	0.6974
	1.0411	2.1961	0.7344	0.7363		1.0576	1.7988	0.7521	0.7783
	1.0404	2.0573	0.8375	0.8467		1.0591	1.7782	0.8552	0.8201
DAY-100									
298.15 K	0.3547	2.4391	0.1426	0.4434	318.15 K	0.3669	1.7795	0.1993	0.5248
	0.3600	2.3737	0.2917	0.5339		0.3681	1.7533	0.3125	0.6156
	0.3651	2.3564	0.5264	0.6901		0.3667	1.7631	0.4428	0.6945
	0.3700	2.2637	0.6882	0.7732		0.3664	1.7531	0.5937	0.7436
	0.3735	2.2565	0.7979	0.8231		0.3715	1.7389	0.7591	0.8246
	0.3631	2.1741	0.8859	0.8911		0.3731	1.7474	0.8366	0.8915
	1.0463	2.6677	0.1409	0.3694		1.0458	2.1342	0.1537	0.4156
	1.0469	2.4686	0.3117	0.4939		1.0458	2.0999	0.1758	0.4498
	1.0484	2.5328	0.4484	0.5787		1.0478	1.9739	0.3158	0.5692
	1.0486	2.3146	0.5666	0.6823		1.0450	2.0121	0.4507	0.6771
	1.0499	2.3117	0.7059	0.7257		1.0486	1.9532	0.5627	0.6943
	1.0505	2.2644	0.8308	0.8114		1.0483	1.8954	0.7035	0.7876
						1.0512	1.8868	0.8301	0.8628

Simplex–Nelder–Mead method (*7*). The function was defined as

$$F = \sum_{j=1}^{N}(\gamma_j^{\text{exptl}} - \gamma_j^{\text{calcd}})^2 \tag{5}$$

where N = number of experimental data points and γ^{calcd} = calculated activity coefficient, and γ^{exptl} = experimental activity coefficient.

There are some difficulties in applying a G^{E} model, originally developed for real vapor–liquid equilibria, to an adsorbate phase mixture (*12*). But excellent correlations of the experimental data are obtained as shown in Figures 4 and 5, and therefore this way was chosen here. The Tóth isotherm parameters of Table 2 were used for the RAST correlations (*4*) as well as for the predictions with IAST (*3*).

As expected, the toluene + 1-propanol mixture adsorption systems behave nonideally on all three DAY adsorbents, but exhibit large differences according to the degree of dealumination. Generally, at low values of y_1 the aromatic hydrocarbon is adsorbed stronger, which changes at higher y_1 values more or less dramatically. While a distinct adsorption azeotrope is formed on DAY-13, mixture adsorption on DAY-55 and DAY-100 showed only slight real behavior. This observation leads to the conclusions that nonidealities of adsorbate phase behavior are essentially caused by surface heterogeneity, e.g., surface hydrophobicity. With an increasing Si/Al ratio the surface of Y-zeolite becomes more and more homogeneous, so that a nearly ideal behavior on DAY-100 is obtained.

If the correlation results are compared with the IAST predictions, for DAY-55 and DAY-100 it can be found, that—for gas phase compositions lower than 0.30—satisfying results are obtained by applying the ideal theory. The observed deviations between IAST and RAST on DAY-13 as a function of the gas mole fraction of toluene are not very surprising. The IAS theory is not able to describe adsorption azeotropic behavior of an adsorbate phase mixture, because real effects are not taken into account. If a system shows an adsorption azeotrope, the IAS theory at least predicts an equilibrium composition curve near the diagonal line. Therefore, the separation factor for toluene calculated by the IAS theory shows a distinct but reasonable trend on all three adsorbents. With an increasing Si/Al ratio the separation factor increases, for example, from 1.41 on DAY-13 to 3.64 on DAY-100.

Conclusions

If it can be accepted that the nonidealities of adsorbate phase behavior are caused by surface hydrophobicity (*12, 13*), then the following can be concluded. With an increasing Si/Al ratio the surface of Y-zeolite becomes more homogeneous, so that a predictive model based on ideal assumptions (IAST) can be used with moderate success. However, not only the aluminum content causes the heterogeneity. As it is shown in the equilibrium composi-

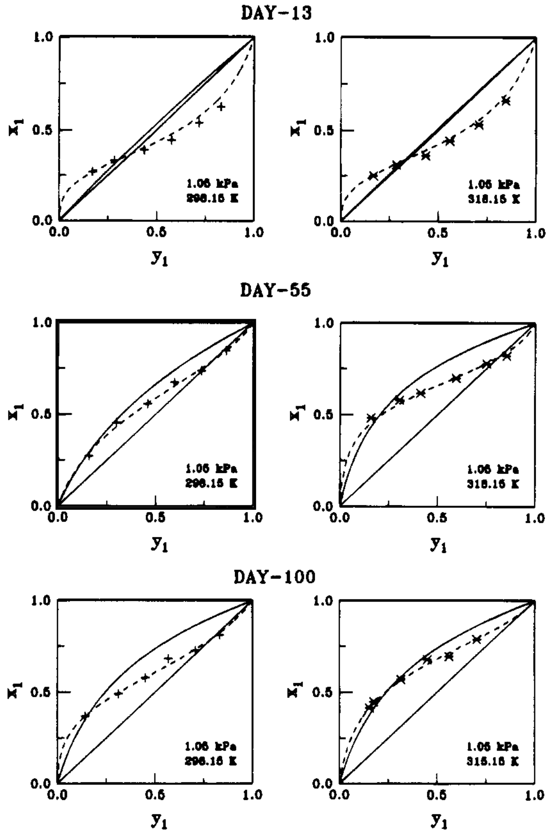

Figure 5. Binary adsorption equilibrium diagrams of toluene (1) + 1-propanol (2) on DAY-13, DAY-55 and DAY-100 at 1.05 kPa and 298.15 and 318.15 K (+, *, experimental values; –, IAST prediction; - - -, RAST correlation).

tion diagram of DAY-100 in Figures 4 and 5 there is still a residual heterogeneity left, which is responsible for the slight real behavior. Perhaps this is caused by different adsorption sites in the windows and supercages of the Y-zeolites. Li and Talu (*14*) named this a structural heterogeneity. They found similar results for binary adsorption equilibria of aromatic hydrocarbons on silicalite, the aluminum-free analogue of ZSM-5. Also a comparison of the IAST prediction with the RAST correlation method illustrates that the information on surface hydrophobicity is almost completely taken into account by the pure adsorption isotherms.

Theoretical work for developing a predictive model to describe multicomponent adsorption equilibria is in progress (*15*).

Acknowledgment

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