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Vapor−Liquid Equilibria and Densities for Propyl Butanoate + Normal Alcohols at 101.32 kPa

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Isobaric vapor−liquid equilibria were measured for propyl butanoate + ethanol, + 1-propanol, and + 1-butanol at 101.32 kPa using a dynamic still with circulation in both phases. After nonideality of the vapor phase was accounted for, the systems studied were thermodynamically consistent and they were correlated by different equations. No azeotrope was found in these mixtures. The predictions of the activity coefficients and of the vapor phase compositions show moderate agreement with the ASOG model and a recent version of UNIFAC. Densities of the above mixtures were also determined at 298.15 K.

Introduction

As a continuation of a long-term study on the thermodynamic properties of liquid mixtures containing alkyl esters, this paper reports isobaric vapor−liquid equilibria (VLE) at 101.32 kPa and densities at 298.15 K for propyl butanoate + ethanol, + 1-propanol, and + 1-butanol mixtures. There appear to be no previous studies on these systems.

In previous papers (Galván et al., 1994; Ortega and Galván, 1994) VLE results on other propyl esters with the same normal alcohols were presented. Good estimations of the activity coefficients and of the vapor phase compositions were achieved with the ASOG model; however, the predictions made with different versions of UNIFAC were not as good. As a general rule, the discrepancies between the experimental and predicted values increase as the chain length of the alkyl ester increases.

Experimental Section

Materials. Propyl butanoate (purum >99 mol %) and normal alcohols (purisss p.a. >99.5 mol %) were from Fluka, and all were used without further purification. However, before use they were degassed by ultrasound and then dried using Union Carbide type 3 Å molecular sieves (Fluka) and kept in dark bottles after this treatment. The experimental densities, ϱ, refractive indexes, $n(D)$, and boiling temperatures, $T_{b,i}$, were measured for all substances, and the values for the n-alcohols were in agreement with those published in a previous paper (Galván et al., 1994); however, for propyl butanoate we found the following values: $\varrho(298.15\ K) = 868.07\ kg{\cdot}m^{-3}$ (868.2; TRC, 1969), $n(D, 298.15\ K) = 1.3975$ (1.3976; TRC, 1969), and $T_{b,1} = 416.65$ K (416.45; TRC, 1969).

Apparatus and Procedure. An all-glass equilibrium still constructed by us was used for the determination of the vapor−liquid equilibria. The details of the still and its operation have been described elsewhere (Ortega et al., 1986). The temperature T and pressure p were measured by digital instruments with an accuracy of ±0.01 K and ±0.02 kPa, respectively. Vapor phase, y_i, and liquid phase, x_i, compositions were determined by densimetry using an Anton-Paar Model DMA-55 densimeter with a precision of $\pm 0.02\ kg{\cdot}m^{-3}$. In this work, the compositions of the vapor and liquid samples were obtained through the correlation of the excess volumes versus ester concentration, and the experimental values and correlations are given in the following section. The accuracies in the mole fraction determined from the relationship $V^E = V^E(x)$ for the two phases were better than ±0.0005.

Table 1. Densities and Excess Volumes at 298.15 K for the Binaries Propyl Butanoate (1) + *n*-Alcohols (2)

x_1	ϱ/ ($kg{\cdot}m^{-3}$)	$10^9 V^E$/ ($m^3{\cdot}mol^{-1}$)	x_1	ϱ/ ($kg{\cdot}m^{-3}$)	$10^9 V^E$/ ($m^3{\cdot}mol^{-1}$)
Propyl Butanoate (1) + Ethanol (2)					
0.000 00	784.96	0.0	0.582 47	848.88	113.8
0.070 02	798.08	20.1	0.654 12	852.89	113.0
0.142 61	809.26	38.2	0.733 53	856.87	106.0
0.179 47	814.17	48.0	0.784 36	859.24	94.1
0.299 08	827.50	75.2	0.879 17	863.29	63.9
0.401 01	836.47	95.9	0.946 66	865.93	33.8
0.485 03	842.67	109.4	1.000 00	867.82	0.0
Propyl Butanoate (1) + 1-Propanol (2)					
0.000 00	799.58	0.0	0.590 63	849.30	139.5
0.121 41	813.90	43.6	0.688 60	854.34	140.5
0.165 38	817.47	61.8	0.749 91	857.27	131.6
0.196 18	821.28	71.8	0.835 77	861.13	105.6
0.230 17	824.39	78.8	0.899 04	863.81	77.1
0.271 21	827.89	91.2	0.912 60	864.40	64.5
0.297 89	830.06	97.7	0.969 77	866.72	25.3
0.385 75	836.65	114.8	1.000 00	867.82	0.0
0.463 57	841.85	126.9			
Propyl Butanoate (1) + 1-Butanol (2)					
0.000 00	805.98	0.0	0.596 39	848.58	164.9
0.066 26	811.85	41.0	0.701 63	854.07	153.8
0.128 50	817.21	71.7	0.741 60	856.06	144.1
0.205 89	823.44	98.8	0.823 17	859.97	115.8
0.231 25	825.34	111.0	0.873 37	862.27	94.9
0.279 29	828.88	123.0	0.915 89	864.16	74.8
0.314 25	831.33	133.6	0.950 40	865.68	53.0
0.459 89	840.75	160.9	1.000 00	867.82	0.0
0.510 74	843.77	164.6			

Table 2. Coefficients *k* and A_i in Eq 1 and Standard Deviations $s(V^E)$

mixture	k	A_0	A_1	$10^9 s(V^E)$/ ($m^3{\cdot}mol^{-1}$)
propyl butanoate (1)				
+ ethanol (2)	1.43	271.7	403.3	0.9
+ 1-propanol (2)	3.04	392.5	594.6	2.3
+ 1-butanol (2)	14.06	601.0	844.7	2.2

Results and Conclusions

Densities. The densities, ϱ, and excess volumes, V^E, were determined for each of the propyl butanoate (1) + n-alcohol (2) systems at (298.15 ± 0.01) K before measuring

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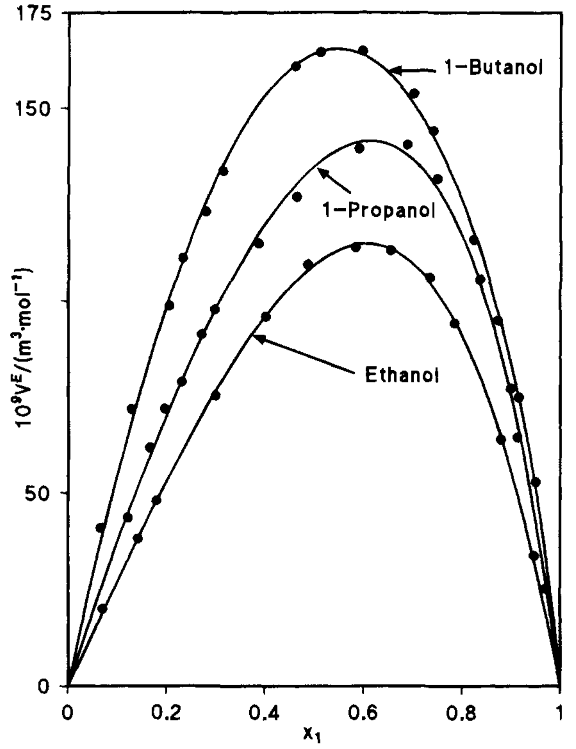

Figure 1. Representation of experimental points (●) and the corresponding correlation curves of V^E at 298.15 K for propyl butanoate (1) + *n*-alcohols (2).

the vapor–liquid equilibrium. The results are given in Table 1. Table 2 presents the values for the coefficients k and A_i in the polynomial equation

$$10^9V^E/(m^3{\cdot}mol^{-1}) = x_1x_2\sum A_i(x_1/[x_1 + k(1 - x_1)])^i \quad (1)$$

used to correlate the V^E values by a method of least squares. Figure 1 plots the curves and the experimental values for each of the mixtures, all demonstrating an expansion effect that increased with alcohol chain length. No literature V^E values were found for the mixtures considered here. The regular distribution pattern for the points (x_1, V^E) indicated that the density measurements were good. Accordingly, as mentioned previously, there were no significant discrepancies between the equilibrium concentration estimates calculated using densities or excess volumes.

Table 3. Experimental Vapor Pressures, $p_i°$, of Propyl Butanoate as a Function of Temperature *T*

T/K	$p_1°$/kPa	*T*/K	$p_1°$/kPa	*T*/K	$p_1°$/kPa
389.70	46.99	410.65	88.16	423.52	124.80
391.10	49.20	411.65	90.68	424.29	127.38
392.50	51.31	412.33	92.48	424.89	129.31
394.20	54.40	412.70	93.43	425.57	131.55
395.79	57.17	415.78	101.45	426.08	133.31
398.09	61.32	416.70	104.11	426.63	135.26
400.45	65.50	417.58	106.57	427.15	136.82
402.22	68.97	418.28	108.69	427.62	138.69
404.26	73.29	418.88	110.41	428.27	140.86
404.95	74.90	418.98	110.68	428.87	143.10
405.85	76.83	419.75	113.05	429.53	145.51
406.76	78.86	420.61	115.58		
407.21	79.94	421.33	117.70		
408.59	83.26	422.11	120.30		
409.68	85.73	422.83	122.60		

Table 4. Coefficients *A*, *B*, and *C* for Eq 2 Used in This Work

	A	*B*	*C*	$s(p_i°)^a$/kPa	ref
propyl-butanoate	5.972 73	1331.41	80.07	0.07	this work
	6.324 82	1543.16	59.45		Fárková and Wichterle (1993)
ethanol	7.113 02	1513.02	55.15		Ortega et al. (1990)
1-propanol	6.869 85	1434.94	74.98		Ortega et al. (1990)
1-butanol	6.917 01	1572.51	70.04		Susial and Ortega (1993)

[a] $s(p_i°) = (\sum(p_i°(\mathrm{exp}) - p_i°(\mathrm{cal}))^2/N)^{1/2}$ where *N* is the number of experimental points.

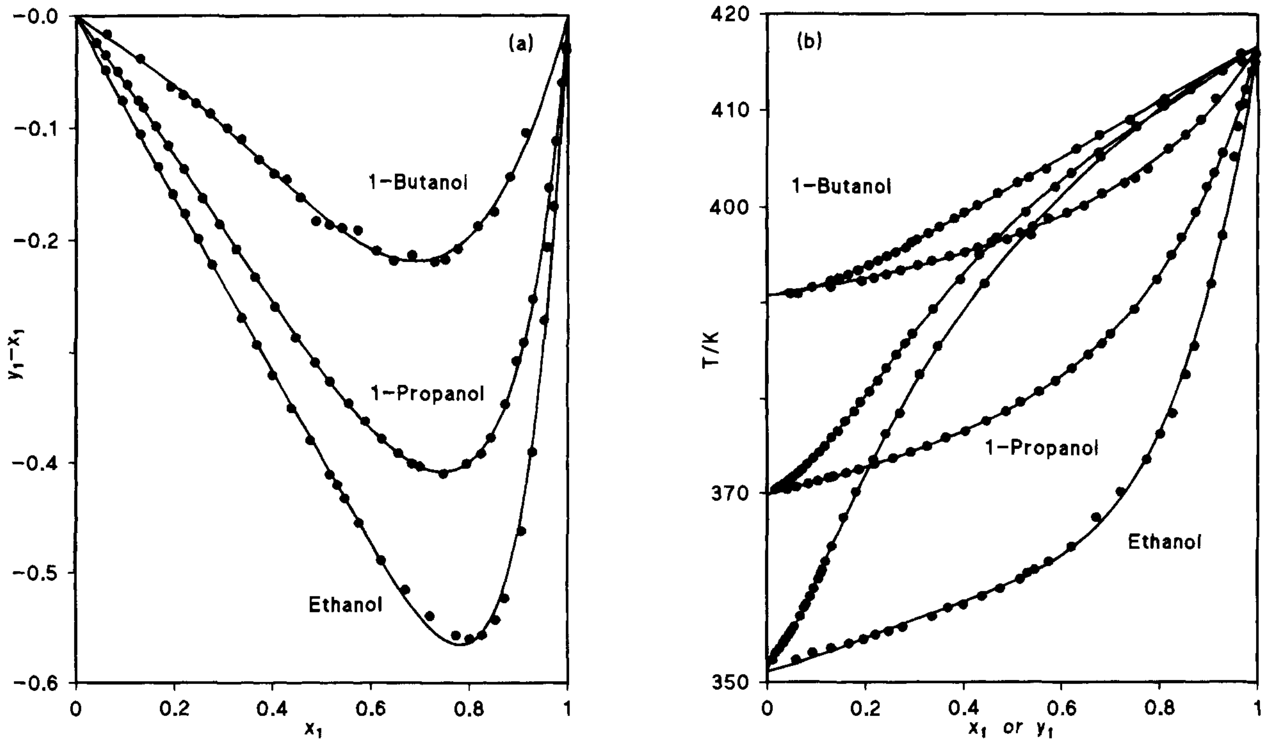

Figure 2. Experimental (●) and calculated values for the mixtures propyl butanoate (1) + *n*-alcohols (2) of (a) $y_1 - x_1$ vs x_1 and (b) *T* vs x_1 or y_1.

Table 5. Experimental VLE Data at 101.32 kPa for Propyl Butanoate (1) + *n*-Alcohols (2)

T/K	x_1	y_1	γ_1	γ_2	T/K	x_1	y_1	γ_1	γ_2
Propyl Butanoate (1) + Ethanol (2)									
351.13	0.0000	0.0000		1.000	361.88	0.5460	0.1129	1.096	1.323
352.35	0.0594	0.0109	1.401	1.004	362.75	0.5746	0.1194	1.067	1.360
353.11	0.0931	0.0174	1.384	1.005	364.35	0.6209	0.1318	1.028	1.424
353.58	0.1305	0.0249	1.388	1.023	367.45	0.6709	0.1555	1.004	1.436
354.08	0.1671	0.0324	1.383	1.040	370.15	0.7204	0.1812	0.991	1.499
354.53	0.1970	0.0376	1.337	1.055	373.55	0.7735	0.2167	0.981	1.586
354.98	0.2215	0.0446	1.386	1.063	376.25	0.8013	0.2412	0.962	1.607
355.39	0.2483	0.0496	1.354	1.079	378.50	0.8267	0.2702	0.969	1.653
355.84	0.2771	0.0553	1.329	1.097	382.50	0.8536	0.3108	0.948	1.636
356.95	0.3364	0.0670	1.271	1.134	385.45	0.8718	0.3488	0.948	1.617
357.85	0.3687	0.0750	1.254	1.144	391.95	0.9051	0.4426	0.948	1.552
358.22	0.4002	0.0796	1.209	1.182	397.09	0.9280	0.5375	0.964	1.475
359.05	0.4379	0.0877	1.180	1.214	405.22	0.9519	0.6804	0.945	1.233
359.88	0.4760	0.0959	1.151	1.253	410.67	0.9714	0.8009	0.940	1.129
360.88	0.5161	0.1049	1.118	1.297	415.85	0.9964	0.9653	0.965	1.383
361.55	0.5310	0.1102	1.114	1.299	416.65	1.0000	1.0000	1.000	
Propyl Butanoate (1) + 1-Propanol (2)									
369.83	0.0000	0.0000		1.000	381.85	0.5879	0.2255	1.020	1.239
370.40	0.0407	0.0167	1.606	1.007	383.15	0.6210	0.2424	0.996	1.262
370.65	0.0594	0.0245	1.600	1.009	384.55	0.6552	0.2633	0.980	1.288
370.99	0.0837	0.0341	1.562	1.013	385.72	0.6833	0.2821	0.970	1.315
371.29	0.1035	0.0421	1.543	1.016	386.69	0.6996	0.2956	0.963	1.318
371.63	0.1252	0.0498	1.491	1.020	389.31	0.7479	0.3375	0.949	1.358
371.74	0.1355	0.0537	1.480	1.024	392.42	0.7946	0.3935	0.947	1.384
372.15	0.1615	0.0628	1.431	1.031	394.99	0.8246	0.4325	0.929	1.401
372.53	0.1874	0.0715	1.386	1.039	396.85	0.8442	0.4667	0.927	1.401
373.10	0.2191	0.0821	1.335	1.048	399.50	0.8736	0.5268	0.936	1.416
373.73	0.2573	0.0944	1.279	1.063	402.13	0.8962	0.5877	0.945	1.392
374.33	0.2924	0.1063	1.241	1.078	403.55	0.9111	0.6197	0.942	1.439
374.97	0.3259	0.1176	1.206	1.092	405.65	0.9287	0.6761	0.951	1.440
375.85	0.3641	0.1310	1.167	1.106	408.35	0.9580	0.7514	0.952	1.740
376.55	0.4040	0.1444	1.132	1.134	410.45	0.9616	0.8075	0.963	1.391
377.65	0.4466	0.1596	1.091	1.154	412.15	0.9747	0.8626	0.970	1.439
378.70	0.4867	0.1770	1.072	1.176	414.07	0.9881	0.9280	0.979	1.524
379.70	0.5166	0.1903	1.051	1.187	415.05	0.9952	0.9676	0.988	1.657
380.75	0.5548	0.2088	1.037	1.216	416.65	1.0000	1.0000	1.000	
Propyl Butanoate (1) + 1-Butanol (2)									
390.85	0.0000	0.0000		1.000	397.28	0.5156	0.3288	1.057	1.093
390.93	0.0622	0.0460	1.485	0.989	397.95	0.5413	0.3515	1.055	1.092
391.59	0.1300	0.0919	1.391	0.992	398.82	0.5733	0.3815	1.055	1.089
392.25	0.1920	0.1290	1.295	1.002	399.45	0.6110	0.4012	1.022	1.134
392.56	0.2171	0.1463	1.287	1.004	400.15	0.6462	0.4277	1.009	1.165
392.92	0.2425	0.1646	1.282	1.003	401.45	0.6834	0.4697	1.010	1.159
392.37	0.2717	0.1848	1.267	1.003	402.55	0.7295	0.5103	0.996	1.210
393.90	0.3070	0.2072	1.237	1.007	403.05	0.7510	0.5336	0.998	1.233
394.38	0.3355	0.2257	1.215	1.010	403.98	0.7765	0.5687	1.002	1.235
394.85	0.3719	0.2433	1.165	1.028	406.05	0.8179	0.6302	0.995	1.221
395.29	0.4031	0.2617	1.141	1.041	407.47	0.8514	0.6763	0.987	1.255
395.75	0.4290	0.2825	1.142	1.042	409.03	0.8829	0.7386	0.996	1.229
396.30	0.4563	0.2937	1.098	1.058	411.15	0.9137	0.8092	0.997	1.144
396.60	0.4883	0.3048	1.056	1.096	416.65	1.0000	1.0000	1.000	

Vapor Pressures. The experimental vapor pressure values for the pure liquid components (T, p_i°), together with the correlation for those values, has a considerable effect in the treatment and analysis of VLE data. Therefore, using the same equilibrium still, p_i° values were determined experimentally for the components employed in this study, and the experimental values were then correlated using the Antoine equation:

$$\log(p_i^\circ/\mathrm{kPa}) = A - B/[(T/\mathrm{K}) - C] \qquad (2)$$

The constants A, B, and C were calculated by a nonlinear regression method, and the corresponding values for n-alcohols were quasi-identical to those published previously (Ortega et al., 1990; Susial and Ortega, 1993). Therefore, Table 3 lists the experimental values for propyl butanoate only; Table 4 presents the values of the Antoine constants used for each of the components in this study.

VLE Data. Treatment and Prediction

The isobaric equilibrium data at (101.32 ± 0.02) kPa measured for the propyl butanoate (1) + *n*-alcohol (2) systems appear in Table 5, which also contains the values of the activity coefficients, γ_i, calculated taking the vapor phase to be nonideal, using

$$\gamma_i = (\phi_i y_i p / x_i \phi_i^\circ p_i^\circ) \exp[(p_i^\circ - p)V_i^{\mathrm{L}}/RT] \qquad (3)$$

in which the fugacity coefficient, ϕ_i and ϕ_i°, values were calculated by

$$\phi_i = \exp[(p/RT)(2\sum_j y_i B_{ij} - \sum_i \sum_j y_i y_j B_{ij})] \qquad (4)$$

The molar volume, V_i^{L}, values and variations in those values with temperature were calculated using a modified version of Rackett's equation (see Spencer and Danner, 1972). The correlations proposed by Tsonopoulos (1974) were used to calculate the virial coefficient values as well

Table 6. Coefficients and Standard Deviations Obtained by Correlating the Dimensionless Function g^E/RT *vs* x_1 using Different Equations in the Binaries Propyl Butanoate (1) + *n*-Alcohols (2)

			$s(g^E/RT)$
	Propyl Butanoate (1) + Ethanol (2)		
Margules	$A_{12} = 0.927$	$A_{21} = 0.414$	0.029
Van Laar	$A_{12} = 1.556$	$A_{21} = 1.529$	0.034
Wilson	$A_{12} = 0.361$	$A_{21} = 1.109$	0.031
NRTL ($\alpha = 0.47$)	$\tau_{12} = -0.136$	$\tau_{21} = 1.056$	0.030
UNIQUAC	$\tau_{12} = 0.581$	$\tau_{21} = 1.118$	0.030
eq 5 ($k = 32.5$)	$A_0 = 0.794$	$A_1 = -3.619$	0.015
	Propyl Butanoate (1) + 1-Propanol (2)		
Margules	$A_{12} = 0.868$	$A_{21} = 2.117$	0.015
Van Laar	$A_{12} = 3.213$	$A_{21} = 2.769$	0.028
Wilson	$A_{12} = 0.159$	$A_{21} = 1.797$	0.022
NRTL ($\alpha = 0.47$)	$\tau_{12} = -0.357$	$\tau_{21} = 1.023$	0.026
UNIQUAC	$\tau_{12} = 1.323$	$\tau_{21} = 0.553$	0.021
eq 5 ($k = 8.44$)	$A_0 = 0.642$	$A_1 = -1.712$	0.003
	Propyl Butanoate (1) + 1-Butanol (2)		
Margules	$A_{12} = 0.416$	$A_{21} = 0.234$	0.004
Van Laar	$A_{12} = 3.435$	$A_{21} = 3.273$	0.006
Wilson	$A_{12} = 0.476$	$A_{21} = 1.307$	0.004
NRTL ($\alpha = 0.47$)	$\tau_{12} = -0.134$	$\tau_{21} = 0.517$	0.005
UNIQUAC	$\tau_{12} = 0.979$	$\tau_{21} = 0.909$	0.004
eq 5 ($k = 3.03$)	$A_0 = 0.351$	$A_1 = -0.220$	0.003

Table 7. Percent Mean Error, $\bar{e}(y_1)$,[a] Obtained in Prediction of the Vapor Phase Mole Fraction, y_1, Using Different Group-Contribution Models and Considering Different Interaction Pairs

	ASOG	UNIFAC-1				UNIFAC-2	
mixture	*OH/COO*[b]	*COH/COO*[c]	*CCOH/COOC*[d]	*OH/COOC*[e]	*OH/COO*[f]	*OH/COOC*[g]	*OH/COOC*[h]
propyl butanoate+							
+ ethanol	13.8	23.8	19.2	28.9	18.3	24.2	3.8
+ 1-propanol	8.5	18.5	16.0	21.6	13.3	13.1	8.7
+ 1-butanol	2.5	12.2	10.6	13.2	6.8	2.7	10.7

[a] $\bar{e}(y_1) = \sum_i(|y_1(\mathrm{exp}) - y_i(\mathrm{cal})|/y_1(\mathrm{exp}))100/N$. [b] Kojima and Tochigi (1979). [c] Fredenslund et al. (1975). [d] Fredenslund et al. (1977). [e] Gmehling et al. (1982). [f] Mecedo et al. (1983). [g] Larsen et al. (1986). [h] Gmehling et al. (1993).

as in the corresponding point-to-point test subroutine published by Fredenslund et al. (1977). The results of that test procedure showed the three mixtures considered in the present study to be consistent.

The equilibrium data were analyzed by correlating the nondimensional function g^E/RT on x_1 by means of various equations, e.g., the Margüles, Van Laar, Wilson, NRTL, and UNIQUAC equations and an expression similar to eq 1 above, used earlier to correlate the VLE data. Equation 1 can be rewritten as follows in a more general form:

$$Q = z(1 - z)\sum A_i(z/[z + k(1 - z)])^i \qquad (5)$$

where z is the variable considered in each case and Q is a general function constrained so that it passes through the end points (0, 1) of z as well through other intermediate points over the interval. The results of fitting the nondimensional function for the excess free energy are shown in Table 6. Equation 5 was also used to correlate the equilibrium compositions and temperatures, setting $Q = y_1 - x_1$ for the former and $Q = T - \sum x_i T_{b,i}$ for the latter. Figure 2a plots the experimental values and the correlation curves for $y_1 - x_1$, and Figure 2b does the same for T. No azeotropes were found for the mixtures considered.

Prediction of the isobaric VLE values was accomplished by using the ASOG model (Kojima and Tochigi, 1979) and the versions of the UNIFAC model published by Fredenslund et al. (1975) (UNIFAC-1), which considers the interaction parameters to be temperature-independent, and by Larsen et al. (1986) and Gmehling et al. (1993) (UNIFAC-2), which takes into account variations in group interaction parameters with temperature. The UNIFAC-1 model as applied was run using all the different possible forms for the alcohol/ester interaction published in the literature. In all cases the predictions were evaluated by comparing the activity coefficients, γ_i, and vapor phase concentrations, y_i, obtained implicitly using eqs 3 and 4. Table 7 lists the mean percentage errors for the y_i estimates; as a general rule, the error decreased with alcohol chain length, except in the UNIFAC-2 model of Gmehling et al. (1993), in which it increased. The different cases of the UNIFAC-1 model and the UNIFAC-2 model of Larsen et al. (1986) yielded the poorest estimates of the VLE values. However, the best predictions obtained using the UNIFAC-1 model were achieved for the interaction pair OH/COO of Macedo et al. (1983), which, oddly, those researchers did not recommend except in special cases. The mean error for the estimates of the activity coefficients for the three mixtures produced by the ASOG model was less than 7%. The mean error for the estimates produced by the UNIFAC-2 version of Gmehling et al. (1993) was less than 10%.

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