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Liquid–Liquid Equilibrium for Ternary Systems of Propyl Vinyl Ether + C_3 or C_4 Alcohols + Water at 298.15 K and Excess Molar Enthalpies for Ternary and Constituent Binary Systems of Propyl Vinyl Ether + Ethanol + Isooctane at 303.15 K

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Liquid–liquid equilibrium (LLE) data for the ternary systems of propyl vinyl ether + C_3 or C_4 alcohols (1-propanol, 2-propanol, 1-butanol, and 2-butanol) + water at 298.15 K were analytically determined by using stirred and thermoregulated cells. Experimental LLE data were correlated with the NRTL and UNIQUAC equations. The distribution and selectivity of C_3 or C_4 alcohols as a solvent were analyzed. Additionally, excess molar enthalpies at 303.15 K are reported for the binary systems of propyl vinyl ether + ethanol, ethanol + isooctane, and propyl vinyl ether + isooctane. These data were obtained by using an isothermal flow calorimeter. The isocline of excess molar enthalpies for the ternary system of propyl vinyl ether + ethanol + isooctane at 303.15 K was calculated using the Radojkovič equation.

Introduction

Alkyl vinyl ethers such as methyl vinyl ether, 1-propyl vinyl ether, isopropyl vinyl ether, 1-butyl vinyl ether, and isobutyl vinyl ether are usually used as industrial solvents and chemical intermediates in the chemical or pharmaceutical industry. Recently, they are used as raw materials for polymer electrolyte membrane fuel cells and as cellulose dyeing assistants. However their reasonable volatility caused significant emissions into the urban atmosphere. Thus, their behavior should be carefully observed and monitored for its effect on the environment. Reliable physical property and phase equilibrium data are required for this purpose, but to date, relatively few investigations have been reported for alkyl vinyl ether compounds. To our knowledge, there are no data for *n*-propyl vinyl ether (PVE) except our previous work.[1–4]

In the present work, we report the liquid–liquid equilibrium (LLE) data for the ternary systems PVE + C_3 or C_4 alcohols (1-propanol, 2-propanol, 1-butanol, and 2-butanol) + water at 298.15 K as a systematic study on the PVE. These data were obtained via an analytical method using a stirred and thermoregulated cell. The experimental ternary LLE data were correlated with NRTL and UNIQUAC models. The distribution and selectivity of C_3 or C_4 alcohols were also analyzed. Additionally, we report the excess molar enthalpies (H^E) at 303.15 K for the binary systems of PVE + ethanol, ethanol + isooctane, and PVE + isooctane. These H^E data were correlated with the Redlich–Kister polynomial. The partial excess molar enthalpies ($\overline{H}_i^{E,\infty}$) were calculated for each binary system with correlated Redlich–Kister parameters. Isoclines of H^E for the ternary system PVE + ethanol + isooctane at 303.15 K were also calculated by using the Radojkovič equation because the prediction of ternary H^E data from constituent binary data is of interest.

Table 1. Purities and Densities of Used Chemicals

		ρ at 298.15 K		UNIQUAC	
chemical	GC analysis	present study	literature value[a]	r^a	q^a
PVE	>99.8	0.76296	–	3.6916[b]	4.6200[b]
ethanol	>99.9	0.78532	0.78500	2.1055	1.9720
1-propanol	>99.9	0.79994	0.79970	2.7799	2.5120
2-propanol	>99.9	0.78132	0.78130	2.7791	2.5080
1-butanol	>99.9	0.80576	0.80600	3.4543	3.0520
2-butanol	>99.9	0.80257	0.80260	3.4535	3.0480
water	>99.9	0.99702	0.99700	0.9200	1.4000
isooctane	>99.9	0.68791	0.68780	5.8463	5.0080

[a] Ref 1. [b] Calculated in this work.

Experimental Section

Materials. The chemicals used in this investigation were of analytical commercial grade purity. Ethanol was provided by J.T Baker chemical Co. PVE, 1-propanol, 2-propanol, 1-butanol, 2-butanol, and isooctane were obtained from Aldrich Co. All the chemicals were dried using pellet type molecular sieves (Fluka Co.) with a pore diameter of 0.4 nm. The purity of the chemicals was checked by gas chromatographic analysis. The water content of the chemicals, determined by a Karl Fischer titratator (Metrohm 684 KF-Coulometer), was less than $7 \cdot 10^{-5}$ $g \cdot g^{-1}$. The wt % of gas chromatographic analysis and the densities of the substance are listed in Table 1 with literature values, UNIQUAC volume *r*, and UNIQUAC surface area parameter *q*.[1] The *r* and *q* parameters for PVE are calculated in this work.

Apparatus and Procedure. LLE measurements have been carried out by measuring the end points of tie-lines for all the ternary systems. The self-designed LLE measuring system used consists of three parts: an equilibrium glass cell, a thermostat

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Table 2. Experimental LLE Data of the Systems PVE (1) + C_3 or C_4 Alcohols (2) + Water (3) at 298.15 K (Mole Fraction)

	organic phase		aqueous phase	
system	x_{11}	x_{21}	x_{13}	x_{23}
PVE (1) + 1-propanol (2) + water (3)	0.9941	0.0000	0.0012	0.0000
	0.9276	0.0512	0.0015	0.0177
	0.8520	0.1118	0.0020	0.0330
	0.7093	0.2188	0.0022	0.0420
	0.5697	0.2998	0.0024	0.0480
	0.4589	0.3542	0.0025	0.0512
	0.3613	0.3983	0.0026	0.0547
	0.2210	0.4244	0.0032	0.0611
	0.1559	0.4107	0.0034	0.0673
	0.1096	0.3884	0.0038	0.0739
PVE (1) + 2-propanol (2) + water (3)	0.9941	0.0000	0.0012	0.0000
	0.8964	0.0673	0.0111	0.0338
	0.7956	0.1416	0.0114	0.0668
	0.6899	0.2111	0.0120	0.0895
	0.5299	0.2985	0.0125	0.1032
	0.3913	0.3496	0.0129	0.1184
	0.2635	0.3666	0.0133	0.1304
	0.1585	0.3463	0.0137	0.1444
PVE (1) + 1-butanol (2) + water (3)	0.9941	0.0000	0.0012	0.0000
	0.8622	0.1062	0.0014	0.0067
	0.6885	0.2273	0.0016	0.0093
	0.5455	0.3227	0.0017	0.0106
	0.4276	0.3878	0.0020	0.0115
	0.3445	0.4311	0.0021	0.0126
	0.2802	0.4570	0.0024	0.0133
	0.1720	0.4953	0.0025	0.0148
	0.1254	0.5092	0.0028	0.0157
	0.1016	0.5123	0.0029	0.0163
	0.0831	0.5168	0.0030	0.0180
	0.0595	0.5206	0.0032	0.0178
(1) + 2-butanol (2) + water (3)	0.9941	0.0000	0.0012	0.0000
	0.9149	0.0591	0.0020	0.0105
	0.8229	0.1191	0.0028	0.0127
	0.6869	0.2040	0.0034	0.0147
	0.5879	0.2640	0.0036	0.0177
	0.4537	0.3301	0.0038	0.0210
	0.3303	0.3873	0.0040	0.0255
	0.2534	0.4168	0.0041	0.0265
	0.1907	0.4330	0.0044	0.0288
	0.1353	0.4203	0.0048	0.0311
	0.0556	0.3999	0.0049	0.0388

(Lauda MD 20 with DLK15 cooler) with a precision temperature measuring system, and a Corning PC-320 magnetic stirrer. The accuracy of the temperature measurement was better than ± 0.02 K. When the system temperature was reached, the sample

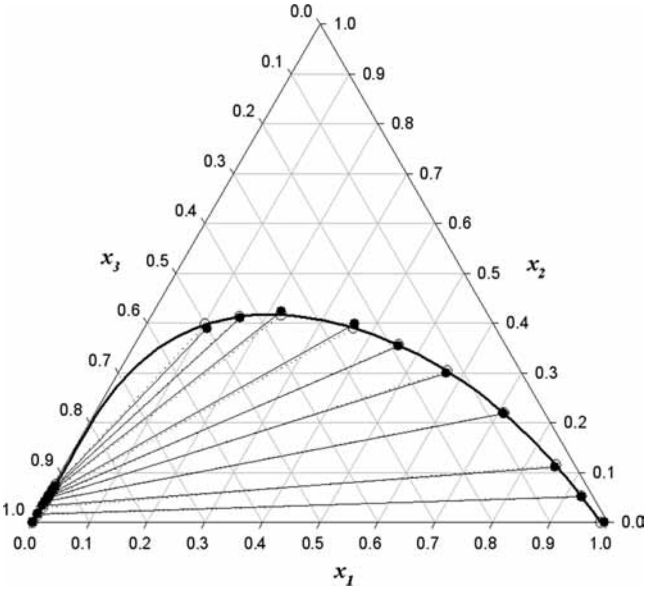

Figure 1. LLE for the ternary system PVE (1) + 1-propanol (2) + water (3) at 298.15 K: ●, experimental value; ○, calculation value. Dashed lines were calculated from the NRTL equation.

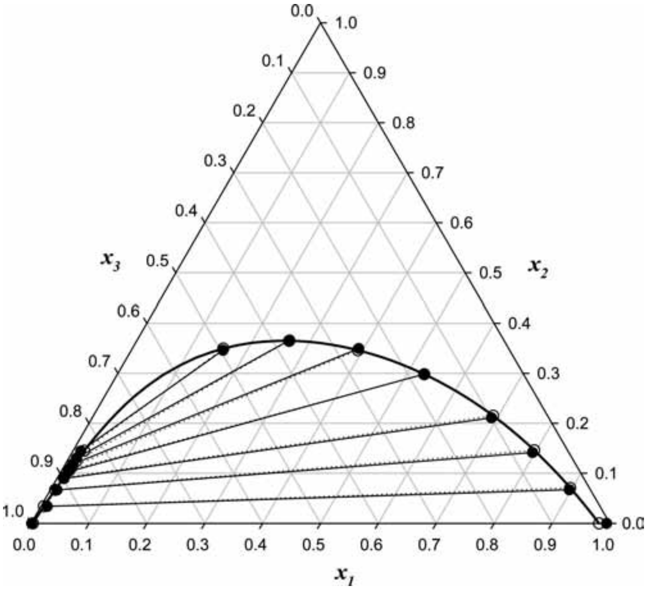

Figure 2. LLE for the ternary system PVE (1) + 2-propanol (2) + water (3) at 298.15 K: ●, experimental value; ○, calculation value. Dashed lines were calculated from the NRTL equation.

Table 3. NRTL and UNIQUAC Model Parameters and rmsd for the Ternary Systems PVE (1) + C_3 or C_4 Alcohols (2) + Water (3) at 298.15 K

		NRTL parameters (*K*)			
system	*i*-*j*	$(g_{ij} - g_{ii})/R$	$(g_{ji} - g_{jj})/R$	α_{ij}	rmsd
PVE (1) + 1-propanol (2) + water (3)	1-2	708.120	−223.430	0.20	0.0030
	2-3	−336.600	1184.300	0.20	
	1-3	805.050	1396.500	0.20	
PVE (1) + 2-propanol (2) + water (3)	1-2	667.240	−220.740	0.20	0.0040
	2-3	−376.570	1080.100	0.20	
	1-3	692.680	1293.100	0.20	
PVE (1) + 1-butanol (2) + water (3)	1-2	8.920	82.076	0.20	0.0029
	2-3	−264.510	1449.400	0.20	
	1-3	709.840	1152.500	0.20	
PVE (1) + 2-butanol (2) + water (3)	1-2	1378.400	−543.600	0.20	0.0047
	2-3	−366.900	1358.000	0.20	
	1-3	640.730	1385.500	0.20	

		UNIQUAC parameters (*K*)		
system	*i*-*j*	$(u_{ij} - u_{ii})/R$	$(u_{ji} - u_{jj})/R$	rmsd
PVE (1) + 1-propanol (2) + water (3)	1-2	457.360	−229.050	0.0041
	2-3	−84.608	275.400	
	1-3	1073.700	657.420	
PVE (1) + 2-propanol (2) + water (3)	1-2	419.360	−215.140	0.0041
	2-3	146.180	2.854	
	1-3	863.940	238.700	
PVE (1) + 1-butanol (2) + water (3)	1-2	21.998	−16.769	0.0014
	2-3	36.674	214.330	
	1-3	994.990	316.320	
PVE (1) + 2-butanol (2) + water (3)	1-2	−106.650	112.340	0.0037
	2-3	−23.011	207.680	
	1-3	867.190	325.460	

mixture was stirred rigorously in an equilibrium cell for about 8 h. It was then allowed to settle for about 16 h at constant system temperature, bringing the mixture into equilibrium. Then, the sample mixture from each liquid phase was taken and analyzed using a gas chromatograph (HP 5890N) equipped with an HP-FFAP (Polyethylene Glycol TPA, 25 m × 0.20 mm × 0.30μm) capillary column and a thermal conductivity detector. The uncertainty of the mole fraction measurement is restricted to $\pm 1 \cdot 10^{-4}$. The procedure is described in detail elsewhere.[5]

A commercial isothermal flow calorimeter (model 7501, Hart Scientific) was used for the measurement of excess enthalpy for binary systems of PVE + ethanol, PVE + isooctane, and ethanol + isooctane. The calorimeter consisted of two solvent pumps (ISCO, LC 2600, 260 cm^3), a temperature-regulated flow cell equipped with a pulsed heater, a Peltier cooler, and a back-pressure regulator to prevent evaporation. The Peltier cooler operated at constant power, producing constant heat loss from the calorimeter cell. The temperature of the cell was kept constant by adjusting the frequency of the pulsed heater to compensate for the cooling from the Peltier cooler and the heat of mixing effect. The uncertainty of the experimental values was estimated to be less than ± 1 % of the H^E values measured. This apparatus has been described in detail.[6]

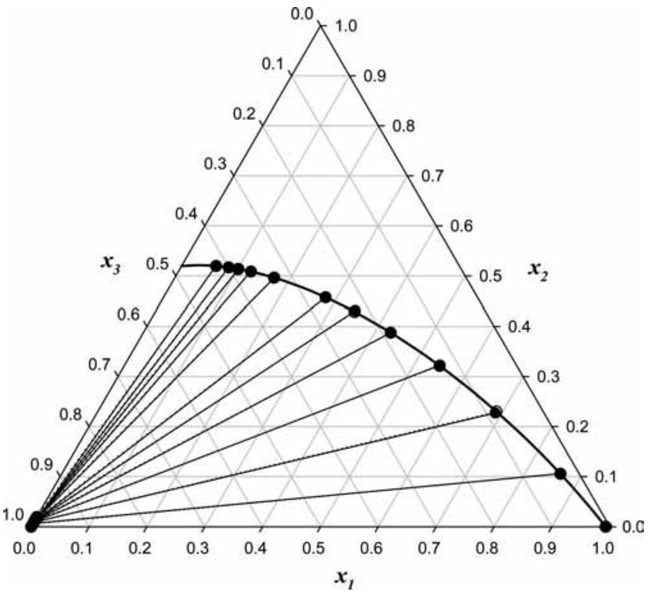

Figure 3. LLE for the ternary system PVE (1) + 1-butanol (2) + water (3) at 298.15 K: ●, experimental value; ○, calculation value. Dashed lines were calculated from the UNIQUAC equation.

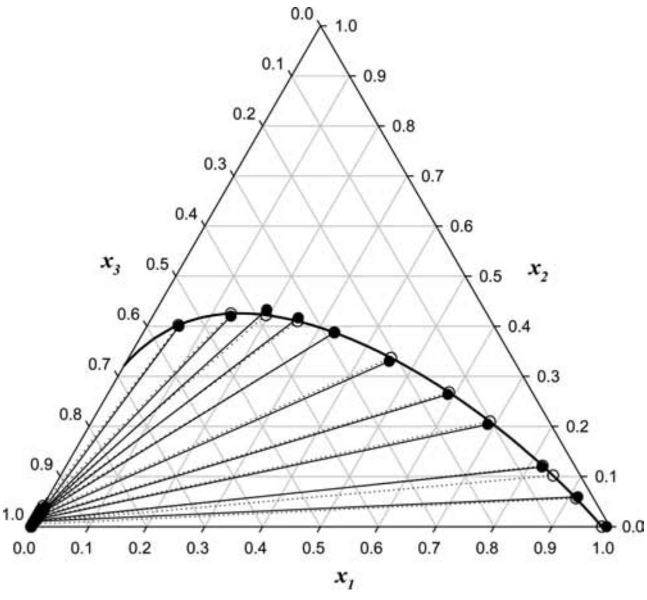

Figure 4. LLE for the ternary system PVE (1) + 2-butanol (2) + water (3) at 298.15 K: ●, experimental value; ○, calculation value. Dashed lines were calculated from the UNIQUAC equation.

Table 4. Calculated Distribution Values (*D*) and Selectivity Values (*S*) for Each Tie-Line

chemical	x_{21}	D	S
1-propanol	0.0512	2.8927	39.8939
	0.1118	3.3879	22.8453
	0.2188	5.2095	17.8812
	0.2998	6.2458	14.4802
	0.3542	6.9180	12.7530
	0.3983	7.2815	11.3709
	0.4244	6.9460	8.8880
	0.4107	6.1025	7.2050
	0.3884	5.2558	5.8803
2-propanol	0.0673	1.9911	19.0060
	0.1416	2.1198	10.2524
	0.2111	2.3587	7.5149
	0.2985	2.8924	6.0759
	0.3496	2.9527	4.7883
	0.3666	2.8113	3.7664
	0.3463	2.3982	2.8109
1-butanol	0.1062	15.8027	114.4969
	0.2273	24.4409	78.3363
	0.3227	30.4434	66.8683
	0.3878	33.7217	58.7951
	0.4311	34.2143	52.0861
	0.4570	34.3609	47.6222
	0.4953	33.4662	40.3171
	0.5092	32.4331	36.9795
	0.5123	31.4294	34.8823
	0.5168	28.7111	31.2193
	0.5206	29.2472	30.9980
2-butanol	0.0591	5.6286	66.0084
	0.1191	9.3780	52.8046
	0.2040	13.8776	44.1724
	0.2640	14.9153	36.0630
	0.3301	15.7190	28.6643
	0.3873	15.1882	22.5884
	0.4168	15.7283	20.9802
	0.4330	15.0347	18.4957
	0.4203	13.5145	15.5541
	0.3999	10.3067	10.8600

Results and Discussion

LLE and Data Correlation. The measured LLE data for the ternary systems PVE + C_3 or C_4 alcohols (1-propanol, 2-propanol, 1-butanol, and 2-butanol) + water at 298.15 K and under atmospheric pressure are listed in Table 2. These measured ternary LLE data were correlated with the NRTL and UNIQUAC equations. The NRTL and UNIQUAC parameters were determined by minimizing the differences between the experimental and calculated mole fractions for each component over all the measured tie lines of the ternary systems. The objective function (OF) used was

$$\mathrm{OF} = \min \sum_i \sum_j \sum_k (x_{ij_k} - x^{\mathrm{c}}_{ij_k})^2 \qquad (1)$$

where x and x^{c} are the experimental and calculated mole fractions, respectively. The subscripts i, j, and k denote component, phase, and tie-line, respectively. The correlated parameters are listed in Table 3, along with the root-mean-square deviation (rmsd) values between experimental and calculated compositions, defined as

$$\mathrm{rmsd} = \left[\frac{\sum_i \sum_j \sum_k (x_{ij_k} - x^{\mathrm{c}}_{ij_k})^2}{6N}\right]^{1/2} \qquad (2)$$

where N is the number of tie-lines. The ternary LLE of each system at 298.15 K are plotted in Figures 1 to 4. The dashed

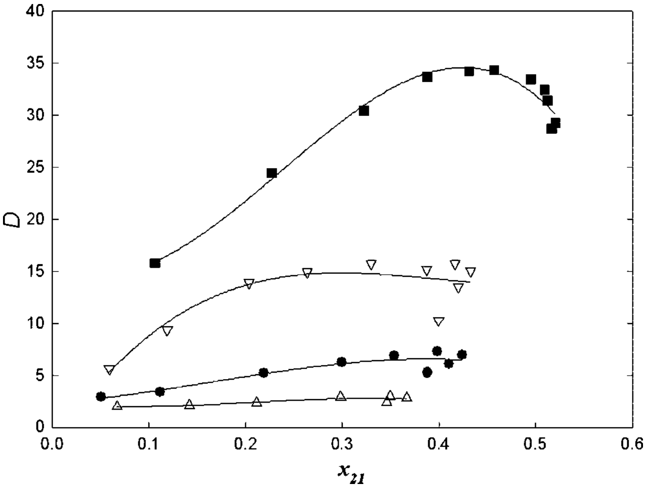

Figure 5. Distribution coefficient (D) against x_{21} for the ternary systems of PVE (1) + C_3 or C_4 alcohols (2) + water (3) at 298.15 K: ●, 1-propanol; ▽, 2-propanol; ■, 1-butanol; △, 2-butanol.

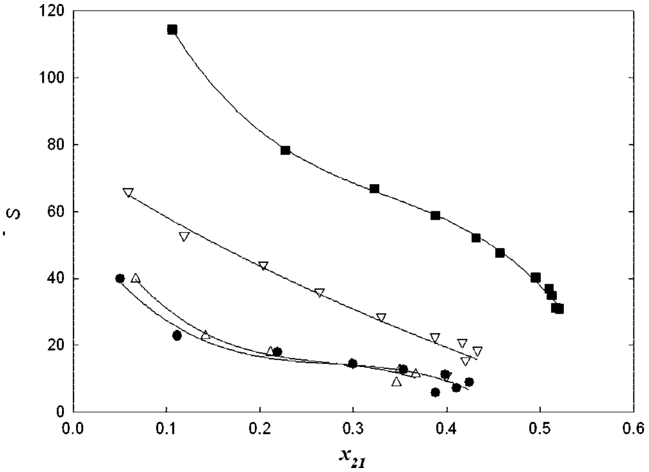

Figure 6. Selectivity (S) against x_{21} for the ternary systems of PVE (1) + C_3 or C_4 alcohols (2) + water (3) at 298.15 K: ●, 1-propanol; ▽, 2-propanol; ■, 1-butanol; △, 2-butanol.

Table 5. Experimental H^{E} Data for the Binary Systems PVE (1) + Ethanol (2), Ethanol (1) + Isooctane (2), and PVE (1) + Isooctane (2) at 303.15 K

x_1	$\frac{H^{\mathrm{E}}}{\mathrm{J\cdot mol^{-1}}}$	x_1	$\frac{H^{\mathrm{E}}}{\mathrm{J\cdot mol^{-1}}}$	x_1	$\frac{H^{\mathrm{E}}}{\mathrm{J\cdot mol^{-1}}}$
		PVE (1) + Ethanol (2)			
0.0265	58.510	0.2564	539.035	0.5468	924.501
0.0543	118.902	0.2973	612.308	0.6080	960.220
0.0836	184.505	0.3409	685.667	0.6741	974.530
0.1145	251.842	0.3873	748.100	0.7456	950.529
0.1470	320.349	0.4368	814.524	0.8231	867.258
0.1814	391.441	0.4899	874.179	0.9076	655.557
0.2178	464.810				
		Ethanol (1) + Isooctane (2)			
0.1291	542.853	0.6525	541.007	0.8679	288.602
0.2383	634.022	0.6974	500.502	0.8942	243.575
0.3320	661.968	0.7380	461.210	0.9185	197.762
0.4132	658.971	0.7749	418.262	0.9410	149.361
0.4842	638.973	0.8086	375.681	0.9620	100.528
0.5469	611.390	0.8395	333.464	0.9817	50.063
0.6026	577.327				
		PVE (1) + Isooctane (2)			
0.0715	106.042	0.4937	429.241	0.7734	317.181
0.1398	194.323	0.5448	430.495	0.8144	275.209
0.2052	265.596	0.5939	423.797	0.8540	228.773
0.2678	324.338	0.6413	405.787	0.8923	175.073
0.3278	367.386	0.6869	383.483	0.9294	116.087
0.3853	399.311	0.7309	353.793	0.9653	57.682
0.4406	419.654				

Table 6. Fitted Parameters for the Redlich–Kister Equation, Standard Deviations, and Partial Molar Excess Enthalpy at Infinite Dilution for Each Binary at 303.15 K

system	A_1	A_2	A_3	A_4	A_5	σ_{st} J·mol^{-1}	$\overline{H}_1^{E,\infty}$ J·mol^{-1}	$\overline{H}_2^{E,\infty}$ J·mol^{-1}
PVE (1) + ethanol (2)	3542.14	1735.64	1142.47	2220.28	1743.54	6.79	2472.23	10384.07
ethanol (1) + isooctane (2)	2532.20	2532.20	1130.61	−1481.18	1613.92	4.12	7463.81	3089.65
PVE (1) + isooctane (2)	1721.52	183.36	26.36	−94.10	−66.69	1.43	1591.93	1770.46

lines are calculated tie-lines using NRTL and UNIQUAC parameters. As shown in the figures, the experimental LLE data agreed very well with the model predictions. Their root-mean-square deviations between calculated and measured values are less than 0.5 % for all measured systems. The slopes of the tie lines presented in the figures show that alcohols (1-propanol, 2-propanol, 1-butanol, and 2-butanol) are more soluble in PVE than in water. The systems PVE (1) + 1-propanol (2) + water (3) and PVE (1) + 2-propanol (2) + water (3) form a miscibility gap of type 1, according to Treybal's classification.[7] Only one binary pair of the PVE + water system shows partial miscibility. The PVE (1) + 1-butanol (2) + water (3) and PVE (1) + 2-butanol (2) + water (3) systems show type 2 phase behavior, because the binary systems PVE + water and 1- or 2-butanol + water show a miscibility gap.

Distribution and Selectivity. In addition, the distribution coefficient (D) of the solute (C_3 and C_4 alcohols) over the two liquid phases in the equilibrium is defined as

$$D = \frac{x_{21}}{x_{23}} \tag{3}$$

where x_{21} is the mole fraction of solute in the PVE-rich phase (organic phase) and x_{23} is the mole fraction of solute in the water-rich phase (aqueous phase). Calculated D values of each solute are presented in Table 4 and plotted in Figure 5. As shown in Figure 5, the D values of 1-butanol are greater than that of 1-propanol, 2-propanol, and 2-butanol among the measured systems. The effectiveness of extraction of water by C_3 or C_4 alcohols could be given by its selectivity (S), which is a measure of the ability of C_3 or C_4 alcohols to act as a separating agent of water from the PVE.

$$S = \frac{x_{21}(x_{21} + x_{31})}{x_{23}(x_{23} + x_{33})} \tag{4}$$

The calculated values of S are also listed in Table 4 and plotted in Figure 6. As shown in Figure 6, selectivity decreases when going through the tie-line end compositions from low concentration to high concentration of C_3 or C_4 alcohols.

Molar Excess Enthalpies. The experimental molar excess enthalpies for the binary systems PVE (1) + ethanol (2), ethanol (1) + isooctane (2) and PVE (1) + isooctane (2), determined at 303.15 K using an isothermal flow calorimeter are listed in Table 5. The measured binary H^E data were correlated with the Redlich–Kister polynomial, eq 5.[8]

$$H_{12}^E/\mathrm{J \cdot mol^{-1}} = x_1 x_2 \sum_{i=1}^{n} A_i (x_1 - x_2)^{i-1} \tag{5}$$

The standard deviation of the fits, σ_{st}, is then defined as

$$\sigma_{st}/\mathrm{J \cdot mol^{-1}} = \left[\frac{\sum_i \left((H^E)_{calcd} - (H^E)_{exptl} \right)_i^2}{(N - n)} \right]^{1/2} \tag{6}$$

where N is the number of experimental data points and n is the number of fitted parameters.

The H^E values of all measured binary systems indicate positive deviations from ideality. The binary H^E data were correlated with the Redlich–Kister polynomial, and the values calculated (solid lines) using the correlated parameters are in good agreement with the experimental values, as shown in

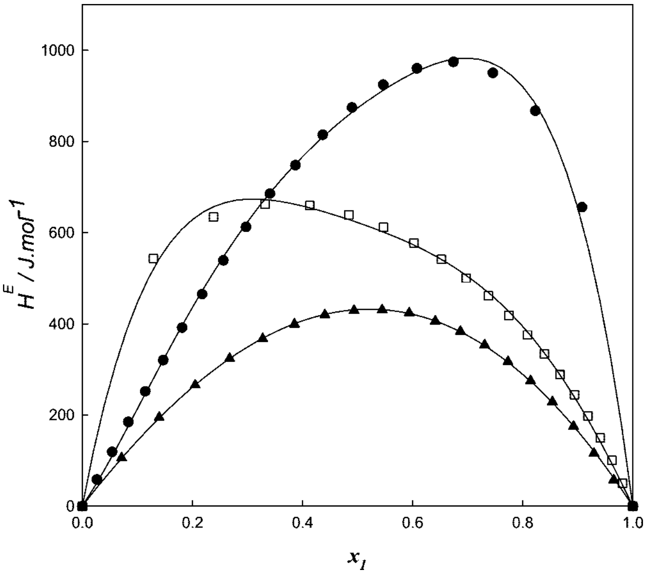

Figure 7. H^E for three binary systems at 303.15 K: ●, PVE (1) + ethanol (2); □, ethanol (1) + isooctane (2); ▲, PVE (1) + isooctane (2). Solid curves are calculated from the Redlich–Kister equation.

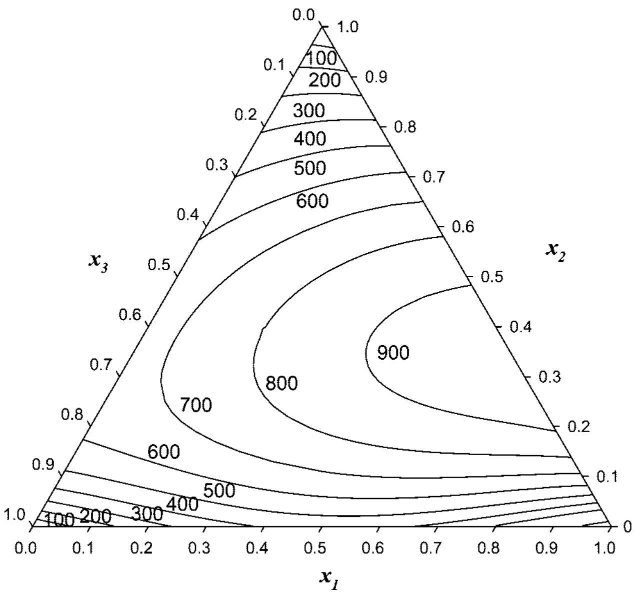

Figure 8. Constant line of H^E at 303.15 K for the ternary system PVE (1) + ethanol (2) + isooctane (3), calculated by the Radojkovič equation.

Figure 7. The adjusted parameters of the Redlich–Kister polynomial are given in Table 6, along with standard deviations between calculated and experimental data. The reliable and extensive data justify the use of four and five parameters. For all systems studied, the obtained standard deviations are within the estimated experimental error of 1 %. The partial excess molar enthalpy at infinite dilution, which can be calculated with fitted Redlich–Kister parameters using eq 7, are also reported in Table 6.

$$\overline{H}_1^{E,\infty}/\mathrm{J\cdot mol^{-1}} = A_1 - A_2 + A_3 - A_4 + A_5$$
$$\overline{H}_2^{E,\infty}/\mathrm{J\cdot mol^{-1}} = A_1 + A_2 + A_3 + A_4 + A_5 \quad (7)$$

Besides, the ternary H^E data can be predicted by the Radojkovič equation,[9] and we calculate ternary H^E data for the system PVE (1) + ethanol (2) + isooctane (3). The Radojkovič equation is used only with the constituent binary Redlich–Kister parameters from Table 6. The predicted isoclines are plotted in Figure 8. As shown in the figure, this ternary system shows positive values of H^E at all the composition ranges as we expected from each constituent binary H^E data.

Conclusion

Liquid–liquid equilibrium (LLE) data for the ternary systems PVE + C_3 or C_4 alcohols (1-propanol, 2-propanol, 1-butanol, and 2-butanol) + water at 298.15 K are reported by measuring the tie-line. The ternary systems, PVE + 1- or 2-propanol + water, have only one partially miscible binary, classified as Treybal's type 1. Meanwhile the systems PVE + 1- or 2-butanol + water have two partially miscible systems, which are classified as Treybal's type 2. They are correlated well with NRTL and UNIQUAC equations. Their root-mean-square deviations are less than 1 %. By the way, 1-butanol showed the largest values of distribution and selectivity for water among the measured systems. Additionally, excess molar enthalpies (H^E) were determined at 303.15 K, for the three binary systems of PVE + ethanol, ethanol + isooctane, and PVE + isooctane. The experimental H^E values for all the binary systems show positive deviations from ideality. These H^E data were correlated well by the Redlich–Kister polynomial. Also, the isoclines of H^E for the ternary system of PVE + ethanol + isooctane at 303.15 K were calculated using the Radojkovič equation. This ternary system shows positive values of H^E at all the composition ranges.

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