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Binary Liquid−Liquid Equilibrium (LLE) for Dibutyl Ether (DBE) + Water from (288.15 to 318.15) K and Ternary LLE for Systems of DBE + $C_1 \sim C_4$ Alcohols + Water at 298.15 K

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The binary liquid−liquid equilibrium (LLE) data for the system dibutyl ether (DBE) + water from (288.15 to 318.15) K and the ternary LLE data for systems of DBE + $C_1 \sim C_4$ alcohols (methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, and 2-butanol) + water at 298.15 K were analytically determined at atmospheric pressure using stirred and thermoregulated cells. The experimental binary and ternary LLE data were correlated with the NRTL and UNIQUAC activity coefficient models. In addition, the distribution and selectivity of $C_1 \sim C_4$ alcohols as solvents were analyzed.

Introduction

Since the use of methyl *tert*-butyl ether (MTBE) as an octane booster in reformulated gasoline causes significant contamination of groundwater, several petroleum and non-petroleum-based compounds (alcohols, other ethers, and aromatics) are studied as substitute material. With reference to their solubility behavior, ether compounds are similar to alcohols but generally less soluble in water. They dissolve easily in most organic compounds and are relatively unreactive, so they are commonly used as organic solvents in industry. For a few decades, some *tert*-ethers have been highlighted as antiknock additives for gasoline, replacing typical leaded compounds which had traditionally been used. However, the phase equilibria and mixture properties of ether compounds such as alkyl or alkyl vinyl ether are not readily available in the literature, even though they are important solvents and excellent extracting agents for use with aqueous systems (owing to their very low water solubility). Therefore, the phase equilibria and mixture properties for several ether compounds have been systematically measured, to provide accurate solubility data. These data could be strongly related to the processing of compounds and to data smoothing by regression using group contribution models.[1,2]

In the present work, we report the binary system liquid−liquid equilibrium (LLE) data for dibutyl ether (DBE) + water at several temperatures from (288.15 to 318.15) K. Also, six ternary system equilibrium LLE data for DBE + $C_1 \sim C_4$ alcohols (methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, and 2-butanol) + water mixtures at 298.15 K and atmospheric pressure are reported as it is not easy to find published LLE data for systems containing DBE. The experimental LLE data for the binary and ternary systems were correlated using two activity coefficient models: NRTL and UNIQUAC. In addition, the distribution and selectivity of $C_1 \sim C_4$ alcohols as solvents were analyzed.

Table 1. Density of Chemicals Used in These Experiments

	ρ/g·cm^{-3} at 298.15 K		GC analysis	UNIQUAC	
chemicals	this work	reference[a]	(wt %)	*r*-value[a]	*q*-value[a]
dibutyl ether	0.76423	0.76410	> 99.9	6.0925	5.1760
methanol	0.78657	0.78660	> 99.9	1.4311	1.4320
ethanol	0.78535	0.78500	> 99.9	2.1055	1.9720
1-propanol	0.79998	0.79970	> 99.9	2.7799	2.5120
2-propanol	0.78135	0.78130	> 99.9	2.7791	2.5080
1-butanol	0.80585	0.80600	> 99.9	3.4543	3.0520
2-butanol	0.80271	0.80260	> 99.9	3.4535	3.0480
water	0.99721	0.99700	> 99.9	0.9200	1.4000

[a] Ref 3.

Table 2. Experimental Data for the Binary System DBE (1) + Water (2)

	organic phase	aqueous phase
T/K	x_1	x_1
288.15	0.9860	0.0000
293.15	0.9832	0.0001
298.15	0.9798	0.0003
303.15	0.9749	0.0007
308.15	0.9682	0.0011
313.15	0.9628	0.0015
318.15	0.9578	0.0020

Experimental Section

Materials. The commercial grade chemicals were used in this investigation without further purification. Ethanol was provided by J.T. Baker Chemical Co. DBE, methanol, 1-propanol, 2-propanol, 1-butanol, and 2-butanol were supplied by Aldrich Co. Water was distilled twice in our laboratory. Methanol was dried using molecular sieves with a pore diameter of 0.3 nm. Other chemicals were dried using sieves of pore diameter of 0.4 nm. The water content of the chemicals, determined by Karl Fischer titration (Metrohm 684 KF-Coulometer), was less than $7 \cdot 10^{-5}$ g·g^{-1}. The purity of the chemicals was checked by gas chromatography and by comparing the density with values reported in the literature.[3] The measured densities and purity of the chemicals are listed in Table 1 with the literature values.

Apparatus and Procedure. The self-designed LLE measuring vessel, similar to that of Horstmann et al.,[4] was used for

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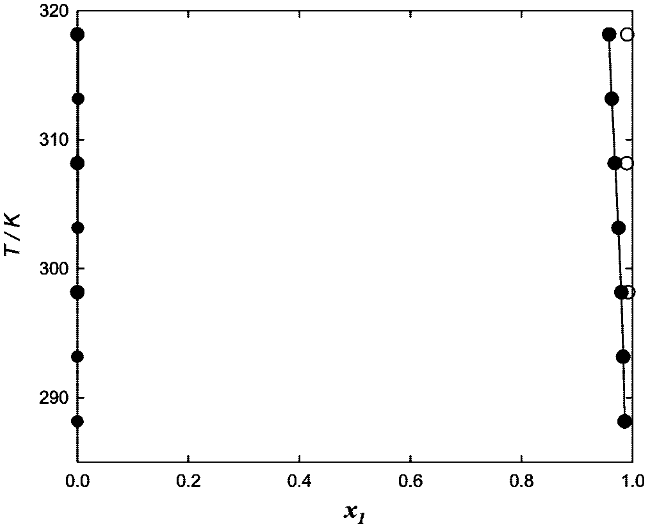

Figure 1. LLE for the binary system DBE (1) + water (2): ●, experimental value; ○, DBE (1) + water (2) by Arce.[6] Solid curves were calculated from the UNIQUAC equation.

Table 3. G^{E} **Model Parameters and Mean Deviation between the Calculated and Experimental Values for the Binary System DBE (1) + Water (2)**[a]

	NRTL		UNIQUAC	
DBE (1) + water (2)	$ij = 12$	$ij = 21$	$ij = 12$	$ij = 21$
A_{ij} (K)	22286.28	121820.54	−9184.22	24892.66
B_{ij}	−131.38	−762.43	64.30	−156.41
C_{ij} (K^{-1})	0.20	1.21	−0.10	0.25
α	0.20		−	
rmsd	0.0005		0.0004	

[a] Parameters (K): $a_{ij} = A_{ij} + B_{ij}T + C_{ij}T^2$.

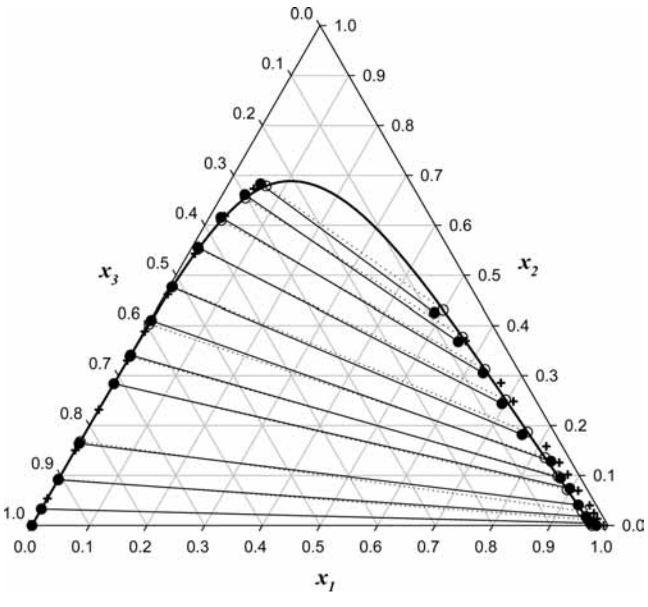

Figure 2. LLE for the ternary system DBE (1) + methanol (2) + water (3) at 298.15 K: ●, experimental value; ○, dashed tie lines were calculated from the UNIQUAC equation; +, DBE (1) + methanol (2) + water (3) by Arce.[6]

Table 4. Experimental LLE Data for the Systems DBE (1) + $C_1 \sim C_4$ Alcohols (2) + Water (3) at 298.15 K

	organic phase		aqueous phase	
system	x_{11}	x_{21}	x_{13}	x_{23}
DBE (1) + methanol (2) + water (3)	0.9694	0.0049	0.0001	0.0329
	0.9529	0.0176	0.0001	0.0914
	0.9279	0.0409	0.0001	0.1642
	0.8965	0.0742	0.0005	0.2839
	0.8700	0.0950	0.0011	0.3411
	0.8368	0.1285	0.0024	0.4102
	0.7598	0.1821	0.0048	0.4766
	0.6939	0.2438	0.0104	0.5558
	0.6304	0.3056	0.0206	0.6157
	0.5562	0.3678	0.0391	0.6613
	0.4862	0.4255	0.0556	0.6829
DBE (1) + ethanol (2) + water (3)	0.9542	0.0098	0.0003	0.0162
	0.9146	0.0288	0.0001	0.0582
	0.8523	0.0758	0.0002	0.1199
	0.7715	0.1392	0.0010	0.1798
	0.6962	0.2047	0.0026	0.2414
	0.6048	0.2605	0.0069	0.3031
	0.4999	0.3354	0.0198	0.3725
	0.3558	0.4118	0.0493	0.4362
	0.2703	0.4503	0.0870	0.4596
DBE (1) + 1-propanol (2) + water (3)	0.9311	0.0292	0.0027	0.0102
	0.7867	0.1612	0.0014	0.0333
	0.6110	0.2798	0.0010	0.0448
	0.4731	0.3696	0.0003	0.0510
	0.3157	0.4459	0.0003	0.0579
	0.1764	0.4756	0.0004	0.0694
	0.0921	0.4401	0.0007	0.0824
	0.0467	0.3678	0.0021	0.0973
DBE (1) + 2-propanol (2) + water (3)	0.9083	0.0460	0.0001	0.0252
	0.7865	0.1322	0.0002	0.0579
	0.5963	0.2666	0.0003	0.0884
	0.3837	0.3891	0.0008	0.1131
	0.2610	0.4333	0.0016	0.1377
	0.1743	0.4345	0.0033	0.1609
	0.0638	0.3547	0.0183	0.2401
DBE (1) + 1-butanol (2) + water (3)	0.7760	0.1391	0.0001	0.0067
	0.6454	0.2541	0.0000	0.0076
	0.5254	0.3341	0.0000	0.0106
	0.3736	0.4202	0.0000	0.0120
	0.2951	0.4763	0.0000	0.0130
	0.1862	0.5216	0.0001	0.0131
	0.0865	0.5354	0.0001	0.0159
	0.0000	0.4995	0.0000	0.0187
DBE (1) + 2-butanol (2) + water (3)	0.8620	0.0775	0.0000	0.0096
	0.7541	0.1699	0.0000	0.0137
	0.5925	0.2936	0.0001	0.0203
	0.3936	0.4219	0.0001	0.0245
	0.2833	0.4729	0.0001	0.0268
	0.2144	0.4996	0.0002	0.0297
	0.1648	0.5098	0.0001	0.0298
	0.1136	0.5137	0.0001	0.0325
	0.0718	0.4974	0.0002	0.0359
	0.0000	0.3105	0.0000	0.0498

the measurement of binary and ternary LLE. The measuring vessel equilibrium temperature was established in a thermostat (Lauda MD 20 with DLK15 cooler) within ± 0.02 K. The temperature of the equilibrium vessel content was monitored using an IBM PC and a temperature measuring system (AΣA F250). The samples in the equilibrium vessel were rigorously stirred using a magnetic stirrer (Corning PC-320), which was equipped with a thermostat. The sample mixture was stirred in its equilibrium vessel for about 6 h and then was allowed to settle for more than 12 h at constant system temperature. After both phases settled, sampling was carefully carried out from the top for the upper and from the bottom for the lower phase while avoiding contamination of both phases during the sampling procedure. The analysis was done using a gas chromatograph (HP 5890N) with an HP-FFAP (Polyethylene Glycol TPA, 25 m × 0.20 mm × 0.30 μm) capillary column and a thermal conductivity detector. We estimate the uncertainty of the calculated mole fraction to be less than ca. $\pm 1 \cdot 10^{-3}$. The procedure is described in detail elsewhere.[5]

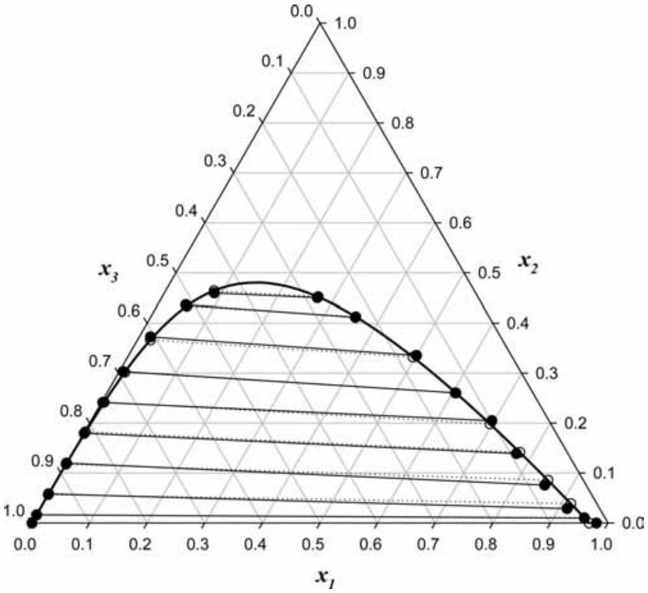

Figure 3. LLE for the ternary system DBE (1) + ethanol (2) + water (3) at 298.15 K: ●, experimental value; ○, dashed tie lines were calculated from the UNIQUAC equation.

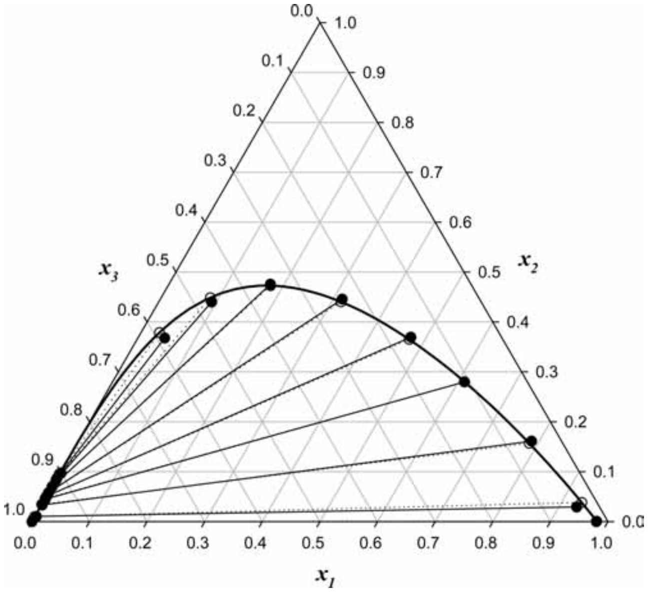

Figure 4. LLE for the ternary system DBE (1) + 1-propanol (2) + water (3) at 298.15 K: ●, experimental value; ○, dashed tie lines were calculated from the UNIQUAC equation.

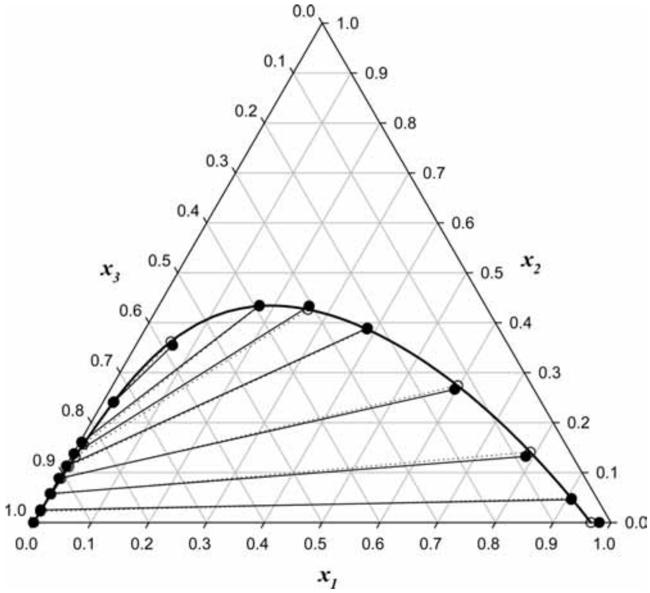

Figure 5. LLE for the ternary system DBE (1) + 2-propanol (2) + water (3) at 298.15 K: ●, experimental value; ○, dashed tie lines were calculated from the NRTL equation.

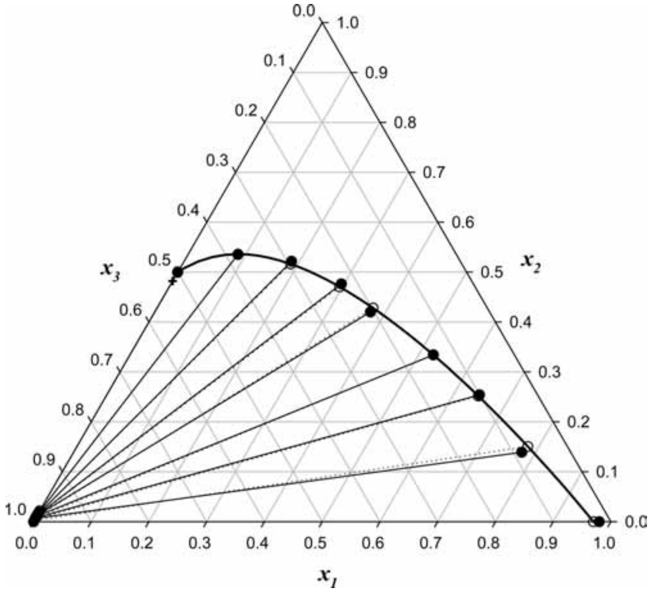

Figure 6. LLE for the ternary system DBE (1) + 1-butanol (2) + water (3) at 298.15 K: ●, experimental value; ○, dashed tie lines were calculated from the UNIQUAC equation; +, 1-butanol (1) + water (3) by Petritis.[9]

Results and Discussion

LLE Data and Data Correlation. The LLE data for the binary system DBE + water at (288.15, 293.15, 298.15, 303.15, 308.15, 313.15, and 318.15) K and atmospheric pressure are listed in Table 2 and plotted in Figure 1 with the literature value of Arce et al.[6] The solubility of DBE in water is negligible. The solubility of water in DBE is significant, and it progressively increases with increasing temperature. The literature data of Arce et al. report, at comparable temperatures, a slightly lower solubility of water in DBE than do our data. The adjustable binary parameters of the NRTL and the UNIQUAC models are listed in Table 3, along with the mean deviations between the experimental values and values recalculated using these two models. Both models gave a good correlation result of less than 0.05 % mean deviation of DBE mole fraction. The solid lines in Figure 1 represent the values calculated using the UNIQUAC model, which gave slightly better correlation results than did the NRTL model.

The measured ternary LLE data for DBE + $C_1 \sim C_4$ alcohols (methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, and 2-butanol) + water mixtures at 298.15 K are given in Table 4. The ternary LLE data for each system are plotted in the form of Gibbs triangles in Figures 2 to 7. Experimental LLE data points were correlated using NRTL and UNIQUAC models. Each constituent binary parameter of both models was determined by minimizing the differences between the experimental and calculated mole fractions for each component over all the measured LLE data of the ternary systems. The objective function (OF) used was

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

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 optimality of the parameters can be evaluated according to the mean deviation in the compositions of coexisting phases.[7] The binary parameters optimized are listed in Tables 5 and 6, with the root-mean-square deviation (rmsd) values between experimental and calculated data, defined as

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

where N is the number of tie lines. The digit number 6 is the number of binary interaction parameters adjusted for a ternary system.

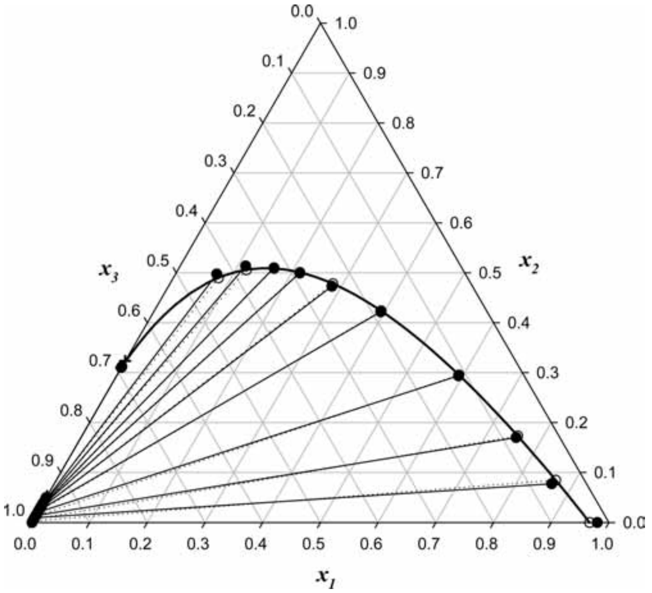

Figure 7. LLE for the ternary system DBE (1) + 2-butanol (2) + water (3) at 298.15 K: ●, experimental value; ○, dashed tie lines were calculated from the UNIQUAC equation; +, 2-butanol (2) + water (3) by Bozdag.[10]

Table 5. NRTL Model Parameters for the Ternary Systems DBE (1) + $C_1 \sim C_4$ Alcohols (2) + Water (3) at 298.15 K

		NRTL parameters (K)			
component 2	$i - j$	$(g_{ij} - g_{ii})/R$	$(g_{ji} - g_{jj})/R$	α_{ij}	rmsd
methanol	1 − 2	−196.39	875.83	0.20	0.0072
	2 − 3	−695.99	406.53	0.20	
	1 − 3	709.68	2958.60	0.20	
ethanol	1 − 2	−307.83	599.10	0.20	0.0052
	2 − 3	−540.35	650.22	0.20	
	1 − 3	568.73	2322.50	0.20	
1-propanol	1 − 2	24.27	285.08	0.20	0.0057
	2 − 3	−272.21	1120.80	0.20	
	1 − 3	650.09	1620.10	0.20	
2-propanol	1 − 2	575.85	−130.18	0.20	0.0049
	2 − 3	−159.20	774.32	0.20	
	1 − 3	525.65	1811.80	0.20	
1-butanol	1 − 2	−652.74	874.78	0.20	0.0047
	2 − 3	−287.34	1443.20	0.20	
	1 − 3	599.99	2303.10	0.20	
2-butanol	1 − 2	−1250.30	2168.50	0.20	0.0074
	2 − 3	−385.98	1367.00	0.20	
	1 − 3	680.49	2402.70	0.20	

Table 6. UNIQUAC Model Parameters for the Ternary Systems DBE (1) + $C_1 \sim C_4$ Alcohols (2) + Water (3) at 298.15 K

	UNIQUAC parameters (K)			
component 2	$i - j$	$(u_{ij} - u_{ii})/R$	$(u_{ji} - u_{jj})/R$	rmsd
methanol	1 − 2	485.67	−55.37	
	2 − 3	−491.47	1206.00	0.0061
	1 − 3	566.95	448.29	
ethanol	1 − 2	262.89	−76.14	
	2 − 3	−303.79	396.13	0.0044
	1 − 3	611.73	234.88	
1-propanol	1 − 2	468.94	−169.89	
	2 − 3	−139.57	394.90	0.0043
	1 − 3	659.22	404.57	
2-propanol	1 − 2	174.00	36.69	
	2 − 3	92.84	138.24	0.0067
	1 − 3	580.72	170.17	
1-butanol	1 − 2	83.80	−81.17	
	2 − 3	14.76	235.90	0.0044
	1 − 3	679.57	150.39	
2-butanol	1 − 2	−61.43	80.91	
	2 − 3	−59.54	258.13	0.0044
	1 − 3	526.83	460.12	

The calculated values in Figures 2 to 7 were obtained using these correlated parameters. For the methanol, ethanol, 1-, and 2-propanol, the hydrogen bonding in water is stronger than that in alcohol since the alcohols are completely miscible in water as shown in Figures 2 to 5. As a consequence, the systems DBE + methanol or ethanol or 1-propanol or 2-propanol + water have one partially miscible binary, classified as Treybal's type I.[8] However, 1- and 2-butanol have a miscibility gap with water because the hydrocarbon portion of the 1- or 2-butanol molecule hardly interacts with water, even though water molecules form strong polar and hydrogen bonds to the hydroxyl group. The systems DBE + 1- or 2-butanol + water are therefore classified as Treybal's type II[8] because they have two partially miscible binaries. The dashed lines are tie-lines calculated using the NRTL or the UNIQUAC model. The experimental and calculated LLE data agreed relatively well as shown in the figures. In the case of the DBE + methanol + water mixture, the experimental data agreed relatively well with literature data of Arce et al.[6] The slopes of the tie lines presented in the figures show that methanol and ethanol are more soluble in water than in DBE. Conversely, 1-propanol, 2-propanol, 1-butanol, and 2-butanol are more soluble in DBE than in water. The correlated parameters of the NRTL and the UNIQUAC model for ternary systems and mean deviations of comparison results

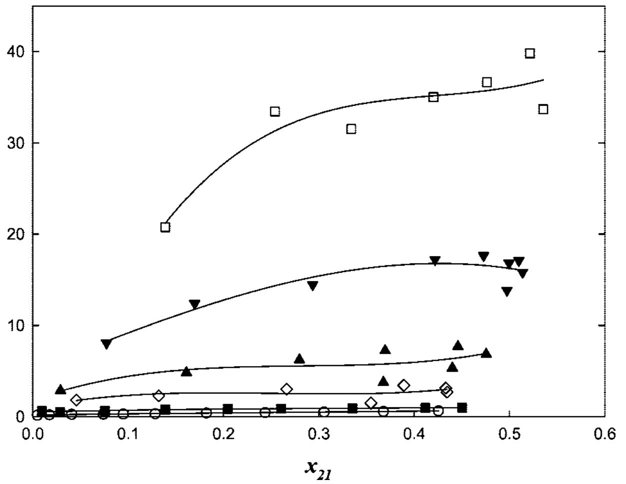

Figure 8. Distribution coefficient (D) against x_{21} for the ternary systems DBE (1) + $C_1 \sim C_4$ alcohols (2) + water (3) at 298.15 K: ○, methanol; ■, ethanol; ▲, 1-propanol; ◇, 2-propanol; □, 1-butanol; ▼, 2-butanol.

Table 7. Experimental Distribution Values *D* and Selectivity Values *S* for each Tie Line

system	x_{21}	D	S
DBE (1) + methanol (2) + water (3)	0.0049	0.1489	4.8667
	0.0176	0.1926	4.0879
	0.0409	0.2491	3.4544
	0.0742	0.2614	2.5240
	0.0950	0.2785	2.1400
	0.1285	0.3133	1.9149
	0.1821	0.3821	1.5830
	0.2438	0.4386	1.4181
	0.3056	0.4963	1.3153
	0.3678	0.5562	1.2042
	0.4255	0.6231	1.1453
DBE (1) + ethanol (2) + water (3)	0.0098	0.6049	13.2043
	0.0288	0.4948	5.7939
	0.0758	0.6322	4.2794
	0.1392	0.7742	3.3848
	0.2047	0.8480	2.7840
	0.2605	0.8595	2.1597
	0.3354	0.9004	1.7648
	0.4118	0.9441	1.3932
	0.4503	0.9798	1.2259
DBE (1) + 1-propanol (2) + water (3)	0.0292	2.8627	41.4371
	0.1612	4.8408	22.6632
	0.2798	6.2455	16.0393
	0.3696	7.2471	13.7500
	0.4459	7.7012	11.2508
	0.4756	6.8530	8.3175
	0.4401	5.3410	5.8787
	0.3678	3.7801	3.9569
DBE (1) + 2-propanol (2) + water (3)	0.0460	1.8254	19.9042
	0.1322	2.2832	10.6922
	0.2666	3.0158	7.4682
	0.3891	3.4403	5.5777
	0.4333	3.1467	4.2512
	0.4345	2.7004	3.2597
	0.3547	1.4773	1.5491
DBE (1) + 1-butanol (2) + water (3)	0.1391	20.7612	92.6746
	0.2541	33.4342	94.2871
	0.3341	31.5189	66.4114
	0.4202	35.0167	55.9014
	0.4763	36.6385	51.9768
	0.5216	39.8168	48.9221
	0.5354	33.6730	36.8578
DBE (1) + 2-butanol (2) + water (3)	0.0775	8.0729	58.4994
	0.1699	12.4015	50.4329
	0.2936	14.4631	35.4886
	0.4219	17.2204	28.3949
	0.4729	17.6455	24.6181
	0.4996	16.8215	21.4081
	0.5098	17.1074	20.4809
	0.5137	15.8062	17.8301
	0.4974	13.8552	14.9239

are given in Tables 5 and 6. The experimental ternary LLE data are correlated well with both the NRTL and the UNIQUAC equations, with the mean deviations less than ca. 0.7 mol % for all the systems.

Distribution Coefficient and Selectivity. The distribution coefficient (D) of the solute ($C_1 \sim C_4$ alcohols) over the two liquid phases in the equilibrium is defined as

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

and the effectiveness of extraction of water by $C_1 \sim C_4$ alcohols could be given by its selectivity (S), which is a measure of the suitability of $C_1 \sim C_4$ alcohols as a separating agent of water from the DBE.

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

x_{21} is the mole fraction of solute in the DBE-rich phase (organic phase); x_{23} is the mole fraction of solute in the water-rich phase (aqueous phase); and x_{31} is the mole fraction of water in the DBE-rich phase. Calculated D and S values of each alcohol are presented in Table 7 and plotted in Figures 8 and 9. As shown in Figures 8 and 9, the D values for 1-butanol are larger than those of methanol, ethanol, 1-propanol, 2-propanol, and 2-butanol in the measured systems. The S value decreases when going through the tie-line end compositions from low to high concentration of $C_1 \sim C_4$ alcohols.

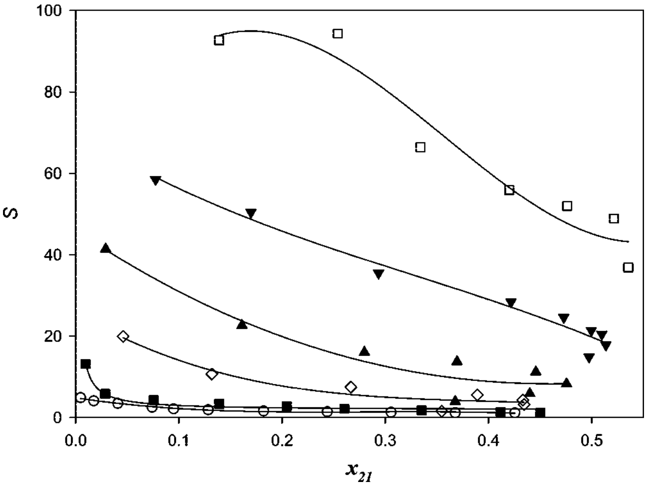

Figure 9. Selectivity (S) against x_{21} for the ternary systems DBE (1) + $C_1 \sim C_4$ alcohols (2) + water (3) at 298.15 K: ○, methanol; ■, ethanol; ▲, 1-propanol; ◇, 2-propanol; □, 1-butanol; ▼, 2-butanol.

Conclusion

The solubility of DBE in water was found to be negligible in the measured temperature ranges, while the solubility of water in DBE was greater than that of DBE in water and the values increased with increasing temperature in the measured temperature ranges. The ternary system of DBE + $C_1 \sim C_3$ alcohols + water is Treybal's type I having a plait point, while DBE + 1- or 2-butanol + water is ternary mixtures of type II having two partially miscible binaries. Methanol is more soluble in water than in DBE, while ethanol, 1-propanol, 2-propanol, 1-butanol, and 2-butanol are more soluble in DBE than in water. Binary and ternary LLE data were correlated well with the NRTL and the UNIQUAC models, with less than 0.7 mol % of rmsd. The distribution coefficient of 1-butanol is larger than those of other alcohols. Selectivity decreased when going through the tie line end compositions from low concentration to high concentration of $C_1 \sim C_4$ alcohols.

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