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Liquid-Liquid Equilibrium for the Quaternary System of o-Xylene(1)+Water(2) +Propionic Acid(3)+1-Butanol(4) at 298.15 K and Atmospheric Pressure

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Abstract−The liquid-liquid equilibrium for the quaternary system of o-xylene(1)+water(2)+propionic acid(3)+1-butanol(4) was measured at 298.15 K and atmospheric pressure. Binodal curves, tie-lines, a distribution curve, and a selectivity curve for the quaternary system have been determined in order to investigate the effect of binary solvents, o-xylene and 1-butanol, on extracting propionic acid from aqueous solution. In addition, these experimental tie line data were also compared with the values predicted by the UNIFAC model. In this study, the UNIFAC model was capable of predicting the compositions of this quaternary system within an average RMSD of 1.13 mol%.

Key words: Liquid-Liquid Equilibrium, Quaternary, Ternary, UNIFAC, Propionic Acid

INTRODUCTION

With an increase in demand for environmentally friendly materials, the recovery of organic acids from dilute solutions resulting from fermentation processes has become of interest. The economics of the process, however, depends on the development of an effective recovery method for the organic acids from the broth [Roychoudhury et al., 1995]. Solvent extraction processes as a promising recovery technique have been proposed as an alternative to the conventional precipitation process [Hartl and Marr, 1993], and many solvents have been tried to improve such recovery [Dramur and Tatli, 1993; Briones et al., 1994; Arce et al., 1995; Fahim and Al-Muhtaseb, 1996; Fahim et al., 1997]. Propionic acid is being used as a cellulosic solvent in the pharmaceutical industry and also can be used to provide propionates, which are used as fungicides [Kirk and Othmer, 1947]. It is also used in the electroplating industry and to prepare perfume esters.

The purpose of this work was to determine LLE data of o-xylene (1)+water(2)+propionic acid(3)+1-butanol(4). The LLE data for the quaternary system were measured at 298.15 K and atmospheric pressure. The effect of the mixed solvents to separate propionic acid from aqueous solution was investigated. The system studied was a type C quaternary system, and the ternary systems relevant to the quaternary mixtures at 298.15 K have been reported for the type 1 systems of o-xylene(1)+water(2)+propionic acid(3) and water(2)+propionic acid(3)+1-butanol(4), and the type 2 system of o-xylene(1)+water(2)+1-butanol(4). The LLE data have also been predicted with the UNIFAC method [Fredenslund, 1977; Gupte and Danner, 1987; Larsen et al., 1987], and the calculated data are compared with the experimental data at 298.15 K.

EXPERIMENTAL SECTION

1. Materials

The o-xylene, 1-butanol, and propionic acid used in this work were supplied by Aldrich with a started purity of 99.5%, and ordinary bidistilled water was used. The purity of each of the components was checked by gas chromatography. The GC analysis did not show any appreciable peaks of impurities for all the components and determined >99.5 mass fraction purities for o-xylene, 1-butanol, and propionic acid. They were used without further purification.

2. Apparatus

The apparatus consisted of a microburet (0.1 mL minimum scale), sample bottle (about 200 mL), and clear water jacket, and was circulated by constant temperature water from a water bath as shown in Fig. 1.

3. Quaternary Equilibrium Data Determination

According to the Prausnitz's classification [Prausnitz et al., 1980], the quaternary system in this study was formed type C, which con-

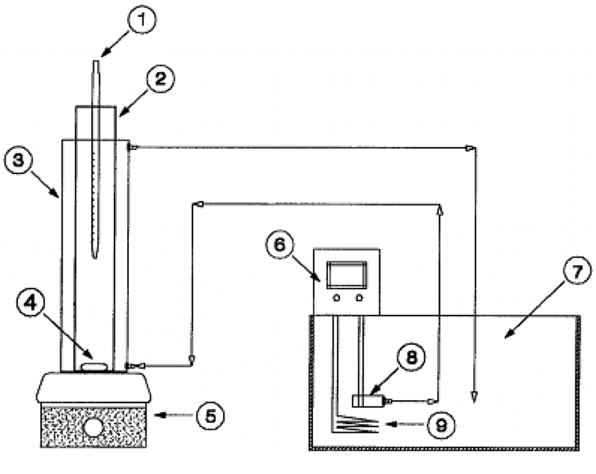

Fig. 1. Schematic diagram of experimental apparatus.

1. Micro buret	6. Temperature controller
2. Sample bottle	7. Water bath
3. Water jacket	8. Circulation pump
4. Magnetic stirring bar	9. Heater
5. Magnetic stirrer	

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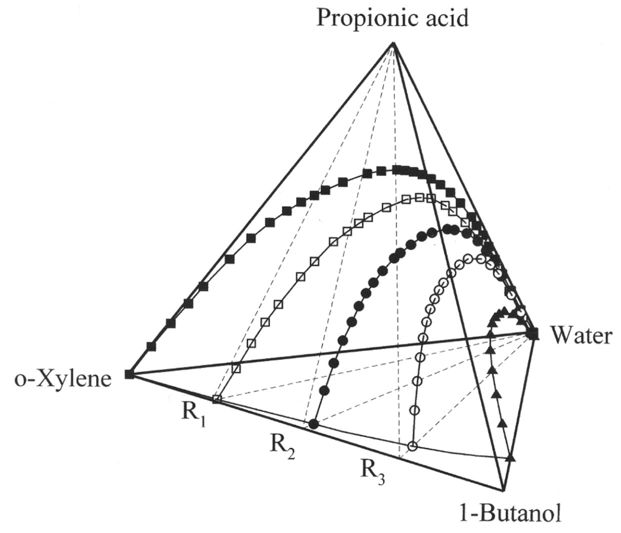

Fig. 2. Phase equilibrium of o-xylene(1)+water(2)+propionic acid(3) +1-butanol(4). R_1, R_2, and R_3 denote quaternary sectional planes.

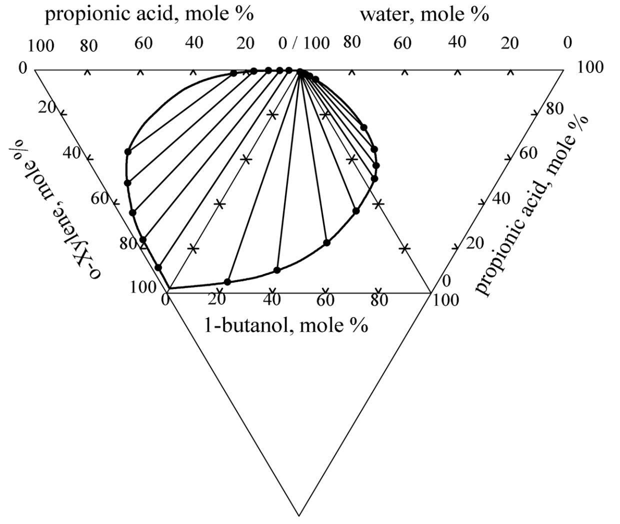

Fig. 3. Binodal curves and tie lines of three ternary mixtures making up o-xylene(1)+water(2)+propionic acid(3), water(2)+propionic acid(3)+1-butanol(4), and o-xylene(1)+water(2)+1-butanol(4) at 298.15 K, respectively.

tains two ternary LLE mixtures of type 1 having a plait point, and one ternary LLE mixture of type 2 having two pairs of partially miscible compounds in Treybal's classification [Sørensen and Arlt, 1980]. The study of the quaternary solubility surface was carried out for a system made up of water, propionic acid, and mixtures of o-xylene and 1-butanol in the ratios of 100/0, 75/25, 50/50, 25/75, and 0/100 (pseudoternary systems) in mass fraction with the following analytical method. Fig. 2 shows the method schematically, where R is the ratio of the solvents.

The mutual solubility (binodal curve) in o-xylene(1)+water(2)+propionic acid(3)+1-butanol(4) was determined at 298.15 K and atmospheric pressure by the cloud-point method as described by Othmer et al. [1941]. For the determination of the mutual solubility data, an accurately known mass of a homogeneous mixture of two compounds was placed in a thermostated glass-stoppered bottle, one of the nonconsolute compounds was titrated into the bottle from a microburet with an uncertainty of ±0.1 mL until the solution became turbid, and the amount titrated was recorded. The bottle was kept in a constant-temperature bath. The temperature of the bath was maintained at (298.15±0.1) K. The end point was confirmed by adding the consolute component until the turbidity disappeared. The procedure was repeated to obtain the phase-boundary curve.

For the tie line measurement, an equilibrium cell was immersed in a thermostat controlled at the desired temperature (±0.1 K). The pure components were added, and the mixture was stirred for at least 1 hour with a magnetic stirrer. The two-phase mixture was allowed to settle for at least 24 hours. Samples were taken by a syringe from the upper and lower mixtures.

The mixtures of aqueous and organic phase were analyzed on an HP 5890 series II gas chromatograph, equipped with a thermal conductivity detector (TCD). A 6 ft×1/8 in column packed with Porapak Q was used. The injector and detector temperatures were maintained at 498.15 K. The column temperature was programmed

Table 1. Binodal curve data for the ternary systems; o-xylene(1)+water(2)+propionic acid(3), water(2)+propionic acid(3)+1-butanol(4), and o-xylene(1)+water(2)+1-butanol(4)

T/K	o-Xylene(1)+Water(2)+Propionic acid(3)			Water(2)+Propionic acid(3)+1-Butanol(4)			o-Xylene(1)+Water(2)+1-Butanol(4)		
	$100x_1$	$100x_2$	$100x_3$	$100x_1$	$100x_2$	$100x_3$	$100x_1$	$100x_2$	$100x_3$
298.15	97.92	2.08	0.00	45.45	0.00	53.55	84.10	3.50	12.40
	76.13	2.96	20.91	47.26	4.80	47.94	74.59	4.78	20.63
	64.07	5.05	30.88	49.91	8.35	41.74	63.30	6.90	29.80
	47.93	10.85	41.22	53.67	10.69	35.64	53.18	10.09	36.73
	39.51	15.18	45.31	59.03	11.71	29.26	41.90	14.70	43.40
	27.23	25.93	46.84	65.29	11.58	23.13	28.13	22.55	49.32
	19.65	35.29	45.06	71.35	10.75	17.90	18.10	30.02	51.88
	9.23	51.08	39.69	80.85	8.26	10.89	0.00	46.45	53.55
	3.59	64.86	31.55	85.74	6.67	7.59	-	-	-
	0.75	79.84	19.41	91.07	4.42	4.54	0.02	99.18	0.80
	0.38	85.13	14.49	94.81	2.54	2.65	0.02	99.16	0.82
	0.07	99.93	0.00	99.07	0.00	0.93	0.03	99.06	0.91

for an initial temperature of 428.15 K and a final temperature of 548.15 K. The heating rate was 348.15 K/min, and the flow rate of the helium carrier gas was 30 mL/min. The uncertainty of the mass fraction measurements for the overall composition determination was ±0.002.

RESULTS AND DISCUSSION

The liquid-liquid equilibrium for the quaternary system of o-xylene (1)+water(2)+propionic acid(3)+1-butanol(4) was measured at 298.15 K and atmospheric pressure. The quaternary experimental data exhibited type C quaternary LLE behavior as shown in Fig. 2 and were composed of two ternary LLE mixtures of type 1 having a plait point (o-xylene(1)+water(2)+propionic acid(3) and water(2)+propionic acid(3)+1-butanol(4)) and one ternary LLE mixture of type 2 having two immiscible binary pairs (o-xylene(1)+water(2)+1-butanol (4)). Fig. 3 shows the experimental results for the ternary mixtures constituting the quaternary mixtures for o-xylene(1)+water(2)+propionic acid(3)+1-butanol(4).

The mutual solubility data and the equilibrium tie line compositions measured at 298.15 K for the ternary mixtures of o-xylene (1)+water(2)+propionic acid(3), water(2)+propionic acid(3)+1-butanol(4), and o-xylene(1)+water(2)+1-butanol(4) system are shown in Tables 1 and 2. Quaternary solubility data concerning the planes R_1(75/25), R_2(50/50), and R_3(25/75) are given in Table 3, and the tie line results of the quaternary mixtures for o-xylene(1)+water(2)+propionic acid(3)+1-butanol(4) system at 298.15 K are given in Table 4. The effect of the mixing ratio on the binodal curves for the quaternary system at 298.15 K is shown in Fig. 4. As shown in Fig. 4, the heterogeneous zone of 1-butanol was increased with the addition of o-xylene, but mixed solvents, o-xylene and 1-butanol in the ratios of 75/25, 50/50, and 25/75 were not better than the pure solvent, o-xylene as an extraction solvent.

Because the most important factor in liquid-liquid extraction is the selectivity of the solvent, the solvents having a higher distribution and selectivity for the solute should be selected after considering the recovery, chemical stability, boiling and freezing points, corrosion, density, and so forth. For those properties, the most important factors are distribution and selectivity. They are shown in the following equations.

Table 2. Liquid-liquid equilibrium compositions for the ternary systems; o-xylene(1)+water(2)+propionic acid(3), water(2)+propionic acid(3)+1-butanol(4), and o-xylene(1)+water(2)+1-butanol(4)

T/K	o-Xylene(1)+Water(2)+Propionic acid(3)					
	Organic phase			Aqueous phase		
	$100x_1$	$100x_2$	$100x_3$	$100x_1$	$100x_2$	$100x_3$
298.15	88.67	2.47	8.86	0.18	96.14	3.68
	76.13	2.96	20.91	0.21	92.63	7.16
	64.07	5.05	30.88	0.24	88.37	11.39
	50.75	9.84	39.41	0.52	82.68	16.80
	36.72	16.95	46.33	1.45	74.61	23.94
T/K	Water(2)+Propionic acid(3)+1-Butanol(4)					
	Organic phase			Aqueous phase		
	$100x_1$	$100x_2$	$100x_3$	$100x_1$	$100x_2$	$100x_3$
298.15	47.03	4.22	48.75	97.87	0.78	1.35
	49.35	7.79	42.86	96.43	1.63	1.94
	53.71	10.69	35.60	94.60	2.66	2.74
	62.57	11.64	25.79	91.56	4.20	4.24
T/K	o-Xylene(1)+Water(2)+1-Butanol(4)					
	Organic phase			Aqueous phase		
	$100x_1$	$100x_2$	$100x_3$	$100x_1$	$100x_2$	$100x_3$
298.15	74.60	4.90	20.50	0.015	99.188	0.797
	53.20	10.20	36.60	0.021	99.156	0.823
	28.20	22.50	49.30	0.023	99.11	0.867
	10.00	36.77	53.23	0.026	99.08	0.912

$$D=\frac{\text{propionic acid mol \% in solvent layer}}{\text{propionic acid mol \% in water layer}}=\frac{x_{31}}{x_{32}} \qquad (1)$$

$$S=\frac{\text{propionic acid mol \% in solvent}-\text{free solvent layer}}{\text{propionic acid mol \% in solvent}-\text{free water layer}}$$

Table 3. Binodal curve data for the o-xylene(1)+water(2)+propionic acid(3)+1-butanol(4) quaternary system

T/K	w(1)/w(4)=75/25				w(1)/w(4)=50/50				w(1)/w(4)=25/75			
	$100w_1$	$100w_2$	$100x_3$	$100x_4$	$100x_1$	$100x_2$	$100x_3$	$100x_4$	$100x_1$	$100x_2$	$100x_3$	$100x_4$
298.15	73.95	1.40	0.00	24.65	47.89	4.22	0.00	47.89	22.90	8.41	0.00	68.69
	60.48	3.23	16.13	20.16	38.87	6.71	15.55	38.87	20.45	10.01	8.18	61.36
	50.63	5.48	27.01	16.88	32.07	10.20	25.66	32.07	18.34	11.96	14.67	55.03
	38.62	10.00	38.50	12.88	27.04	13.47	32.45	27.04	16.55	13.95	19.86	49.64
	32.70	12.79	43.61	10.90	22.25	18.27	37.23	22.25	13.58	18.50	27.17	40.75
	28.81	15.51	46.08	9.60	15.00	28.00	42.00	15.00	10.05	27.22	32.57	30.16
	18.39	24.00	51.49	6.12	12.34	31.92	43.40	12.34	9.02	30.38	33.54	27.06
	13.50	29.00	53.00	4.50	5.75	46.50	42.00	5.75	7.41	35.89	34.46	22.24
	6.00	43.00	49.00	2.00	3.75	52.80	39.70	3.75	4.29	48.16	34.68	12.87
	5.04	46.64	46.65	1.67	2.40	59.10	36.10	2.40	2.50	58.00	32.00	7.50
	3.50	52.97	42.37	1.16	1.69	64.28	32.34	1.69	1.62	65.00	28.50	4.88
	1.88	66.20	31.30	0.62	1.22	75.04	22.52	1.22	0.77	81.00	15.90	2.33
	1.15	98.46	0.00	0.39	1.11	97.78	0.00	1.11	0.64	97.43	0.00	1.93

Table 4. Calculated and experimental liquid-liquid equilibrium compositions for the o-xylene(1)+water(2)+propionic acid (3)+1-butanol(4) quaternary system at 298.15 K

	Organic phase				Aqueous phase			
	$100x_1$	$100x_2$	$100x_3$	$100x_4$	$100x_1$	$100x_2$	$100x_3$	$100x_4$
	x(1)/x(4)=75/25							
Exp.	62.06	7.60	7.29	23.05	0.01	97.83	1.07	1.10
Calc.	60.72	7.44	7.06	24.78	0.01	98.03	1.08	0.88
Exp.	51.05	10.62	19.97	18.36	0.02	95.26	3.55	1.17
Calc.	50.04	10.41	19.38	20.17	0.02	95.45	3.59	0.94
Exp.	41.28	14.49	30.13	14.10	0.04	91.95	6.71	1.29
Calc.	40.52	14.22	29.28	15.98	0.04	92.13	6.79	1.04
Exp.	32.84	19.11	37.75	10.30	0.10	87.35	11.03	1.52
Calc.	32.27	18.78	36.73	12.22	0.10	87.52	11.16	1.22
Exp.	26.22	23.88	42.90	7.00	0.33	80.02	17.66	1.99
Calc.	25.79	23.48	41.78	8.95	0.33	80.20	17.87	1.60
	x(1)/x(4)=50/50							
Exp.	32.69	20.91	6.64	39.76	0.01	97.90	0.84	1.25
Calc.	31.67	20.66	6.50	41.17	0.01	97.97	0.85	1.17
Exp.	26.02	24.89	17.93	31.16	0.02	95.87	2.78	1.33
Calc.	25.23	24.61	17.55	32.61	0.02	95.92	2.81	1.25
Exp.	20.15	29.55	26.67	23.63	0.03	93.31	5.20	1.46
Calc.	19.54	29.23	26.12	25.11	0.03	93.35	5.25	1.37
Exp.	15.01	35.01	32.89	17.09	0.06	89.88	8.39	1.67
Calc.	14.56	34.64	32.22	18.58	0.06	89.90	8.47	1.57
Exp.	10.48	41.58	36.53	11.41	0.15	84.64	13.11	2.10
Calc.	10.17	41.14	35.79	12.90	0.15	84.64	13.24	1.94
	x(1)/x(4)=25/75							
Exp.	12.28	41.43	9.34	36.95	0.01	96.86	1.75	1.38
Calc.	10.36	38.25	10.02	41.37	0.01	96.98	1.51	1.50
Exp.	8.46	40.13	12.10	39.31	0.01	95.80	2.76	1.43
Calc.	8.89	40.46	14.18	36.47	0.01	96.05	2.38	1.56
Exp.	7.71	45.55	15.75	30.99	0.01	94.62	3.87	1.50
Calc.	7.76	42.47	17.83	31.94	0.01	95.01	3.34	1.64
Exp.	6.43	48.10	17.47	28.00	0.02	93.30	5.11	1.57
Calc.	6.70	44.67	20.95	27.68	0.02	93.84	4.42	1.72
Exp.	5.70	53.92	20.25	20.13	0.03	91.79	6.52	1.66
Calc.	5.69	47.10	23.51	23.70	0.03	92.50	5.65	1.82

$$=\frac{x_{31}/(x_{31}+x_{21})}{x_{32}/(x_{32}+x_{22})} \tag{2}$$

Compositions are expressed in mole fraction x_{CL}, where the first index refers to the component (2=water, 3=propionic acid) and the second index refers to the phase (1=upper layer, 2=lower layer).

Figs. 5 and 6 illustrate the effect of the distribution and selectivity for the mixtures in the ratios 75/25, 50/50, and 25/75. As shown in the result of Fig. 4, they were also not better than the pure solvent, o-xylene, as an extraction solvent. Perhaps this is because of the higher mutual solubility of water and 1-butanol when compared with that for water and o-xylene. The results indicate that the solubility of o-xylene is high enough in propionic acid.

1. UNIFAC Predicted LLE Data

Because experimental data are often missing or of poor quality, group contribution methods have become increasingly valuable. The great advantage of the group contribution makes it possible to predict the compositions of a large number of systems using only a relatively small number of group interaction parameters. The most common group contribution method for the prediction of phase equilibrium is UNIFAC.

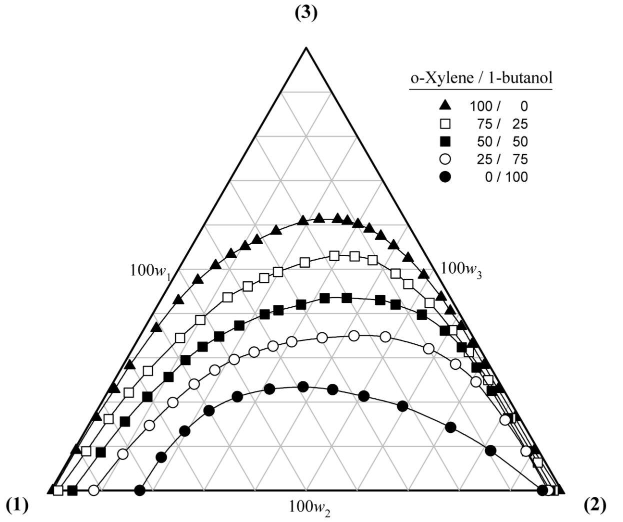

Fig. 4. The effect of the mixing ratio on binodal curves of the o-xylene(1)+water(2)+propionic acid(3)+1-butanol(4) quaternary system at 298.15 K.

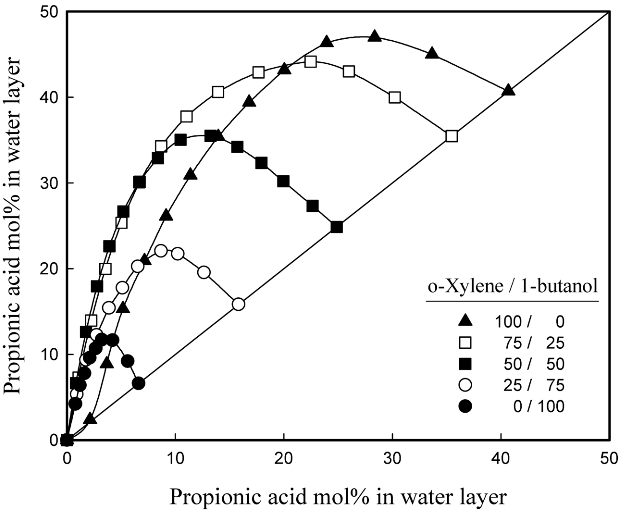

Fig. 5. Distribution of propionic acid between aqueous and organic phases at 298.15 K.

This model requires two group interaction parameters (a_{mn} and a_{nm}) per pair of groups and the group volume and surface areas of individual groups for the estimation of the activity coefficient γ_i. The UNIFAC group volume, surface area, and interaction parameters used in this work are shown in Table 5 [Magnussen et al., 1981].

The experimental LLE data and tie lines were compared with predictions obtained by the UNIFAC. The RMSD (Root Mean Square Deviation), deviation of the estimate by experimental and calculated compositions, was obtained from Eq. (3) for the ternary system and from Eq. (4) for the quaternary system.

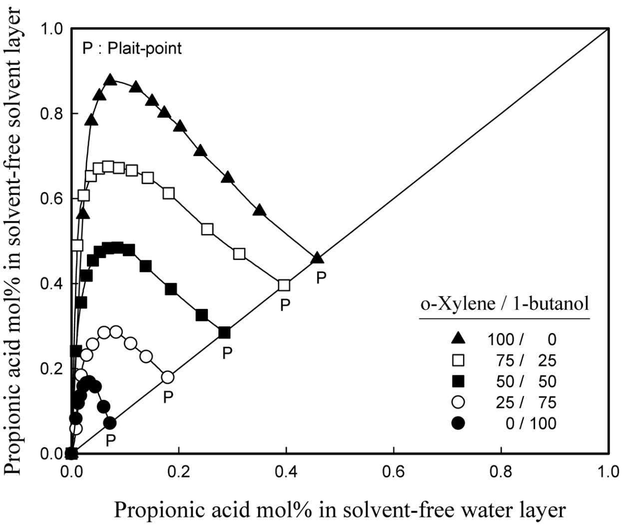

Fig. 6. Selectivity of propionic acid between aqueous and organic phases at 298.15 K.

$$\mathrm{RMSD}_3=100\left[\sum_{i=1}^{N}\sum_{j=1}^{3}\sum_{k=1}^{2}(\mathrm{X}_{jk}^{exp}(\mathrm{i})-\mathrm{X}_{jk}^{cal}(\mathrm{i}))^2/6\mathrm{N}\right]^{1/2} \tag{3}$$

$$\mathrm{RMSD}_4=100\left[\sum_{i=1}^{N}\sum_{j=1}^{4}\sum_{k=1}^{2}(\mathrm{X}_{jk}^{exp}(\mathrm{i})-\mathrm{X}_{jk}^{cal}(\mathrm{i}))^2/8\mathrm{N}\right]^{1/2} \tag{4}$$

where, N is the number of tie line data.

For the ternary systems, the predictions for o-xylene(1)+water (2)+propionic acid(3) and water(2)+propionic acid(3)+1-butanol (4) composed of type 1 were shown within an average RMSD of 2.74 mol%, and the prediction for o-xylene(1)+water(2)+1-butanol (4) composed of type 2 was shown within 1.17 mol%. For the quaternary system, it was shown that the UNIFAC model was capable of predicting the compositions within an average RMSD of 1.13 mol%.

CONCLUSION

Liquid-liquid equilibrium data were determined for the quaternary system of o-xylene(1)+water(2)+propionic acid(3)+1-butanol(4) at 298.15 K and atmospheric pressure. The variation of selectivity with mixtures of both solvents is of little significance, with o-xylene appearing as the better extraction solvent on account of the wide heterogeneous zone in the solubility diagram and the distribution and selectivity curve. The use of mixed solvents in the ratios of 75/25, 50/50, and 25/75 to improve the extraction of propionic acid will be advisable only when both solvents show some favorable properties, such as the distribution coefficient and/or selectivity, in order to take advantage of the synergistic effect of the mixture.

NOMENCLATURE

a_{ij} : interaction parameter for i-j interaction [dimensionless]
Q_k : surface area parameter, for group j [dimensionless]
R_k : volume parameter, for group k [dimensionless]
T : absolute temperature [K]
x_i : mole fraction for component i [%]
γ_i : activity coefficient for component i [dimensionless]

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Table 5. The UNIFAC group volume (R_k), surface area (Q_k), and interaction parameters (a_{nm})

	CH_3	CH_2	OH	H_2O	COOH	ACH	$ACCH_3$	R_k	Q_k
CH_3	0	0	644.6	1300.0	139.4	−114.8	−115.7	0.9011	0.848
CH_2	0	0	644.6	1300.0	139.4	−114.8	−115.7	0.6744	0.5400
OH	328.2	328.2	0	28.73	−104.0	−9.210	1.270	1.0000	1.2000
H_2O	342.4	342.4	−122.4	0	−465.7	372.8	203.7	0.9200	1.4000
COOH	1,744.0	1,744.0	118.4	652.3	0	75.49	147.3	1.3013	1.224
ACH	156.5	156.5	703.9	859.4	461.8	0	167.0	0.5313	0.400
$ACCH_3$	104.4	104.4	4,000.0	5,695.0	339.1	−146.8	0	1.2663	0.968

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