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Liquid−Liquid Equilibria of Water + 2-Butanol + (Methyl Methacrylate or Butyl Methacrylate or Isobutyl Methacrylate) at (288.2 and 318.2) K

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Liquid−liquid equilibrium (LLE) data for the ternary systems water + 2-butanol + methyl methacrylate, butyl methacrylate, and isobutyl methacrylate were determined at atmospheric pressure and at (288.2 and 318.2) K. The reliability of the experimental tie-line data was determined through the Bachman plots. The experimental data were also compared with the values correlated by the NRTL and UNIQUAC models. Good quantitative agreement was obtained with these models. In general, the average deviations from the UNIQUAC model are slightly smaller than those from the NRTL model.

Introduction

Short-chain alcohols are widely used as co-emulsifiers in the emulsion polymerization of many acrylic resins. It is well-known that the influences of the mutual solubility of co-emulsifiers and monomers in water on the results of emulsion polymerization are very large, including the critical micelle concentration (CMC) of emulsifiers, the emulsified nucleation mechanism, and the reaction kinetics, etc. In our laboratory, we are interested in investigating the fundamental solubility properties of those co-emulsifiers and methacrylate monomers in water for the acrylic emulsion polymerization application such as the selections of co-emulsifiers and methacrylate monomers. This paper reports on liquid−liquid equilibria (LLE) for the systems of water + 2-butanol with one of three methacrylate monomers: methyl methacrylate, butyl methacrylate, and isobutyl methacrylate. The phase compositions of two coexistent liquid phases for these investigated systems are measured at (288.2 and 318.2) K and at atmospheric pressure. No literature data were found at comparable conditions. The experimental tie-line data were correlated to test consistency with the Bachman[1] equations. These new LLE data were also correlated with the solution models of the NRTL[2] and the UNIQUAC.[3] Good quantitative agreement was obtained with these models.

Experimental Section

Chemicals. 2-Butanol was obtained from Riedel-deHaën. Methyl methacrylate was supplied by Showa Chemical Co. Ltd. Butyl methacrylate was purchased from Alfa Aesar. Isobutyl methacrylate was obtained from Acros Organics. Deionized distilled water was prepared in our laboratory. The purities of these liquids were greater than 99 %. All the chemicals had been checked by gas chromatography and without further purification before use. No impurity peaks were detected.

Table 1. Average Deviations of GC Calibration Curves

mixture	phase	average deviations[a]
water + methyl methacrylate	organic	0.0016
	aqueous	0.0002
water + butyl methacrylate	organic	0.0019
	aqueous	0.0002
water + isobutyl methacrylate	organic	0.0019
	aqueous	0.0002
water + 2-butanol	organic	0.0012
	aqueous	0.00003

[a] Average deviations = $1/n_{\text{p}} \sum_{j=1}^{n}\text{p}\ |x_{\text{act}} - x_{\text{calb}}|_j$, where n_{p} is the number of calibration points and x is the minor constituent compound. The subscripts "act" and "calb" represent the actual values and calibrated values, respectively.

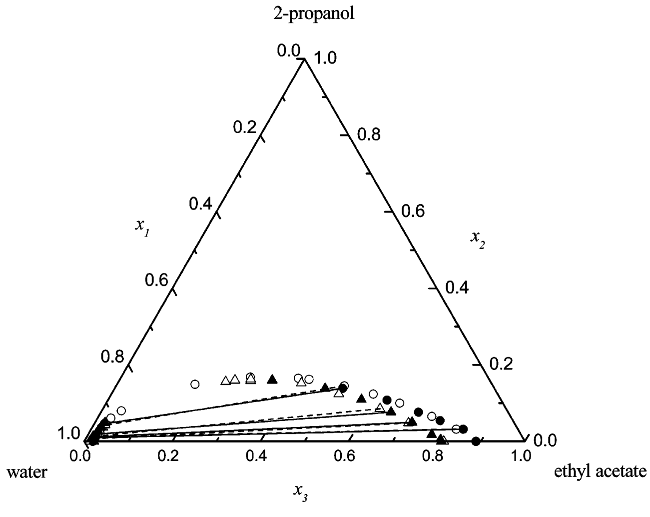

Figure 1. LLE phase diagram for water (1) + 2-propanol (2) + ethyl acetate (3): ○, ref 9 at 283.2 K; Δ, ref 9 at 323.2 K; ●, this work at 283.2 K; ▲, this work at 323.2 K; ----, ref 9 tie-line at 283.2 K or 323.2 K; −, this work tie-line at 283.2 K or 323.2 K.

Apparatus and Procedure. Liquid−liquid equilibrium data for the ternary systems were measured by an apparatus similar to that of Peschke and Sandler,[4] including a jacketed glass cell, a thermostatically controlled bath, a magnetic agitator, and a gas chromatograp. In the jacketed equilibrium glass cell (internal volume of about 20 cm^3), the thermostated water was circulated to control the temperature of the cell within ± 0.1 K. The cell temperature was measured by a precision thermometer (model-1506, Hart Scientific) with a platinum RTD probe to an uncertainty of ± 0.03 K. The prepared mixtures were introduced into the equilibrium cell and were agitated vigorously for at least 3 h to sufficiently mix the compounds and then settled for at least 8 h for complete phase separation. The sample of the

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Table 2. LLE Data for Water (1) + 2-Butanol (2) at Atmospheric Pressure

	this work				literature data				
	organic phase (I)		aqueous phase (II)		organic phase (I)		aqueous phase (II)		
T/K	x_1	x_2	x_1	x_2	x_1	x_2	x_1	x_2	
288.2	0.706	0.297	0.941	0.059	0.7125	0.2875	0.9330	0.0670	ref 5
298.2					0.6971	0.3029	0.9474	0.0526	ref 6
					0.7644	0.2356	0.9664	0.0336	ref 7
					0.6899	0.3101	0.9480	0.0520	ref 8
					0.6768	0.3235	0.9470	0.0530	ref 5
313.2					0.6594	0.3406	0.9582	0.0418	ref 5
318.2	0.675	0.325	0.958	0.042					
323.2					0.6583	0.3417	0.9613	0.0387	ref 5

Table 3. LLE Data for Water (1) + 2-Propanol (2) + Ethyl Acetate (3) at Atmospheric Pressure

	this work						literature data[9]					
	organic phase (I)			aqueous phase (II)			organic phase (I)			aqueous phase (II)		
T/K	x_1	x_2	x_3	x_1	x_2	x_3	x_1	x_2	x_3	x_1	x_2	x_3
283.2	0.112	0.000	0.888	0.981	0.000	0.019	0.111	0.000	0.889	0.981	0.000	0.019
	0.124	0.032	0.844	0.972	0.009	0.019	0.140	0.032	0.828	0.969	0.011	0.020
	0.165	0.054	0.781	0.963	0.018	0.019	0.179	0.065	0.756	0.959	0.020	0.021
	0.203	0.076	0.720	0.953	0.027	0.020	0.234	0.100	0.666	0.949	0.030	0.021
	0.259	0.108	0.633	0.942	0.037	0.021	0.282	0.124	0.594	0.943	0.036	0.021
	0.344	0.139	0.517	0.931	0.046	0.022	0.337	0.145	0.518	0.935	0.042	0.023
							0.409	0.162	0.429	0.926	0.050	0.024
							0.432	0.165	0.403	0.924	0.051	0.025
							0.539	0.168	0.293	0.909	0.061	0.030
							0.674	0.149	0.177	0.876	0.080	0.044
323.2	0.189	0.000	0.811	0.978	0.000	0.022	0.184	0.000	0.816	0.987	0.000	0.013
	0.202	0.018	0.780	0.977	0.007	0.016	0.240	0.048	0.712	0.979	0.007	0.014
	0.231	0.050	0.719	0.972	0.012	0.017	0.286	0.085	0.629	0.973	0.013	0.014
	0.266	0.076	0.658	0.964	0.019	0.017	0.360	0.124	0.516	0.959	0.021	0.020
	0.316	0.109	0.575	0.956	0.026	0.019	0.431	0.152	0.417	0.951	0.027	0.022
	0.384	0.138	0.478	0.943	0.037	0.020	0.543	0.158	0.299	0.941	0.037	0.022
	0.493	0.160	0.346	0.928	0.049	0.023	0.541	0.162	0.297	0.939	0.037	0.024
							0.578	0.160	0.262	0.932	0.042	0.026
							0.601	0.156	0.243	0.931	0.043	0.026

Table 4. LLE Data for Water (1) + 2-Butanol (2) + Methyl Methacrylate (3) at Atmospheric Pressure

	organic phase (I)			aqueous phase (II)		
T/K	x_1	x_2	x_3	x_1	x_2	x_3
288.2	0.056	0.000	0.944	0.997	0.000	0.003
	0.085	0.054	0.862	0.990	0.006	0.004
	0.110	0.099	0.791	0.986	0.010	0.004
	0.165	0.171	0.664	0.981	0.015	0.004
	0.211	0.220	0.569	0.978	0.018	0.004
	0.253	0.257	0.491	0.976	0.020	0.005
	0.307	0.293	0.400	0.974	0.022	0.004
	0.356	0.324	0.320	0.973	0.024	0.004
	0.416	0.347	0.237	0.970	0.028	0.003
	0.504	0.373	0.123	0.963	0.035	0.002
	0.706	0.294	0.000	0.941	0.059	0.000
318.2	0.078	0.000	0.922	0.997	0.000	0.003
	0.109	0.057	0.834	0.993	0.003	0.003
	0.138	0.102	0.761	0.991	0.006	0.003
	0.189	0.173	0.639	0.988	0.009	0.003
	0.231	0.222	0.547	0.986	0.011	0.003
	0.272	0.257	0.472	0.985	0.012	0.003
	0.315	0.294	0.392	0.983	0.014	0.003
	0.364	0.322	0.314	0.981	0.016	0.003
	0.419	0.348	0.233	0.979	0.019	0.003
	0.507	0.373	0.120	0.974	0.025	0.002
	0.675	0.325	0.000	0.958	0.042	0.000

Table 5. LLE Data for Water (1) + 2-Butanol (2) + Butyl Methacrylate (3) at Atmospheric Pressure

	organic phase (I)			aqueous phase (II)		
T/K	x_1	x_2	x_3	x_1	x_2	x_3
288.2	0.051	0.000	0.949	1.000	0.000	0.000
	0.067	0.136	0.797	0.989	0.011	0.000
	0.086	0.207	0.707	0.984	0.016	0.000
	0.115	0.268	0.617	0.980	0.020	0.000
	0.154	0.330	0.516	0.977	0.023	0.000
	0.204	0.388	0.408	0.974	0.026	0.000
	0.247	0.421	0.332	0.972	0.028	0.000
	0.302	0.454	0.244	0.970	0.030	0.000
	0.364	0.469	0.167	0.967	0.033	0.000
	0.444	0.463	0.093	0.962	0.038	0.000
	0.706	0.294	0.000	0.941	0.059	0.000
318.2	0.064	0.000	0.936	1.000	0.000	0.000
	0.082	0.130	0.788	0.994	0.006	0.000
	0.102	0.204	0.695	0.991	0.009	0.000
	0.132	0.267	0.601	0.988	0.012	0.000
	0.170	0.327	0.504	0.986	0.014	0.000
	0.219	0.384	0.397	0.983	0.017	0.000
	0.260	0.420	0.320	0.982	0.018	0.000
	0.321	0.453	0.226	0.980	0.020	0.000
	0.374	0.467	0.158	0.977	0.023	0.000
	0.455	0.456	0.090	0.973	0.027	0.000
	0.675	0.325	0.000	0.958	0.042	0.000

organic-rich phase was carefully taken from the top sampling port of the cell with a syringe, and that of the water-rich phase was taken from a bottom sampling port of the cell. This operating method avoids cross contamination by the other phase during the sampling procedure.

The composition of the sample was analyzed by gas chromatography (GC) (model 9800, China Chromatography Co., Taiwan) with a thermal conductivity detector (TCD) using high purity helium (99.99 %) as a carrier gas. A stainless steel column packed with 10 % Porapak Qs 60/80 (2 m × 1/8″) can clearly separate the constituent compounds of the samples. Five samples were replicated for each phase at a fixed experimental condition, and the area fraction was converted into mole fraction by the calibration equations. Two calibration lines were previously constructed according to the organic-rich or the water-rich phases for each binary system. The deviations of the calibration curves from the actual values are tabulated in Table 1. The experimental mole fractions for each phase were obtained by averaging the results from these five replications. The uncertainty of reported mole fractions was estimated to be less than

Table 6. LLE Data for Water (1) + 2-Butanol (2) + Isobutyl Methacrylate (3) at Atmospheric Pressure

	organic phase (I)			aqueous phase (II)		
T/K	x_1	x_2	x_3	x_1	x_2	x_3
288.2	0.048	0.000	0.952	1.000	0.000	0.000
	0.066	0.134	0.799	0.989	0.011	0.000
	0.087	0.206	0.707	0.984	0.016	0.000
	0.114	0.267	0.619	0.980	0.020	0.000
	0.155	0.329	0.516	0.977	0.023	0.000
	0.211	0.386	0.404	0.975	0.026	0.000
	0.250	0.423	0.328	0.972	0.028	0.000
	0.312	0.452	0.236	0.970	0.030	0.000
	0.370	0.467	0.163	0.968	0.032	0.000
	0.449	0.459	0.092	0.964	0.036	0.000
	0.706	0.294	0.000	0.941	0.059	0.000
318.2	0.074	0.000	0.926	1.000	0.000	0.000
	0.083	0.129	0.788	0.994	0.006	0.000
	0.104	0.202	0.694	0.991	0.009	0.000
	0.131	0.262	0.607	0.989	0.011	0.000
	0.178	0.325	0.498	0.986	0.014	0.000
	0.228	0.382	0.389	0.984	0.016	0.000
	0.266	0.417	0.317	0.982	0.018	0.000
	0.328	0.451	0.222	0.980	0.020	0.000
	0.387	0.457	0.156	0.978	0.022	0.000
	0.457	0.454	0.089	0.973	0.027	0.000
	0.675	0.325	0.000	0.958	0.042	0.000

± 1 %. Because no literature LLE data were available at comparable conditions for those ternary systems above, the LLE measurements of the water + 2-butanol system and the water + 2-propanol + ethyl acetate system were conducted to test the validity of our experimental procedure. Table 2 compares the experimental LLE results of the water + 2-butanol system with the literature values. Furthermore, Figure 1 and Table 3 also compare the experimental LLE results for the ternary system of water + 2-propanol + ethyl acetate with the literature values. They show that our measurements agree with literature values within the experimental uncertainties.

Experimental Results

The LLE measurements were made at temperatures of (288.2 and 318.2) K under atmospheric pressure. Tables 4 to 6 list the experimental results for water + 2-butanol with methyl methacrylate, butyl methacrylate, and isobutyl methacrylate, respectively. The symbol I represents the organic-rich phase, and II represents the aqueous phase. Figures 2 to 4 are the phase diagrams for the ternary systems of water + 2-butanol + methyl methacrylate, water + 2-butanol + butyl methacrylate, and water + 2-butanol + isobutyl methacrylate, respectively. Because 2-butanol + methacrylate is the only liquid pair that is completely miscible and the two liquid pairs (water + 2-butanol) and (water + methacrylate) are partially miscible, all investigated ternary systems behave as Type 2 of LLE. Very small amounts of the organic compounds were found in the aqueous phase, and water dissolved appreciably in the organic-rich phase. The phase diagram shows no plait point. The experimental results show that the area of the two-phase region in the butyl methacrylate system and in the isobutyl methacrylate system is similar in size, whereas that in the methyl methacrylate system is appreciably smaller.

Consistency of Experimental Tie-Line Data. In this study, Bachman correlation eq 1 was used to ensure the quality of the obtained experimental tie-line data.

$$w_{32} = A + B(w_{32}/w_{11}) \qquad \text{Bachman correlation} \qquad (1)$$

where w_{11} is the mass fraction of water in the aqueous phase; w_{32} is the mass fraction of methacrylate in the organic-rich phase; and A and B are the parameters of the Bachman correlation. The correlation parameters and the standard deviations σ were determined by the least-squares method by a Marquardt algorithm. The correlated results are represented in Table 7. The Bachman plot is also shown in Figure 5 for those three investigated systems. From Table 7, the values of standard deviation σ at temperature 288.2 K are as low as 0.0123, 0.0104, and 0.0108 for the methyl methacrylate, butyl methacrylate, and isobutyl methacrylate systems, respectively, and at temperature 318.2 K are 0.0070, 0.0074, and 0.0065, respectively. Furthermore, as seen from Figure 5, the linearity of the plot for those three investigated systems, we revealed that our measured LLE data in this study have a high degree of consistency.

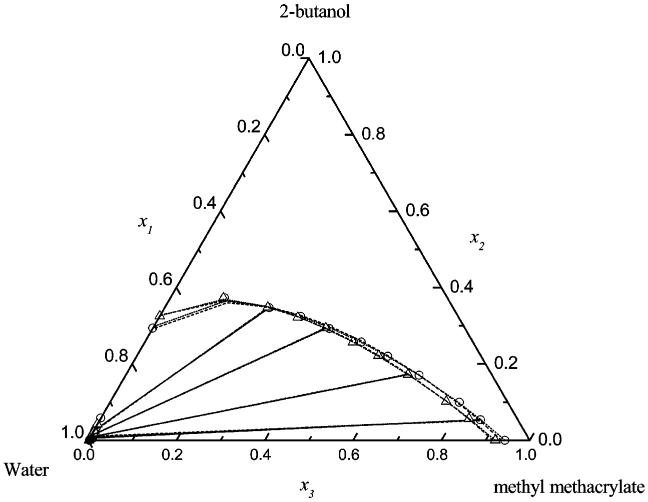

Figure 2. LLE phase diagram for water (1) + 2-butanol (2) + methyl methacrylate (3): ○, exptl data at 288.2 K; Δ, exptl data at 318.2 K; —, exptl tie-line at 288.2 K or 318.2 K; ----, NRTL correlation at 288.2 K or 318.2 K; ····, UNIQUAC correlation at 288.2 K or 318.2 K.

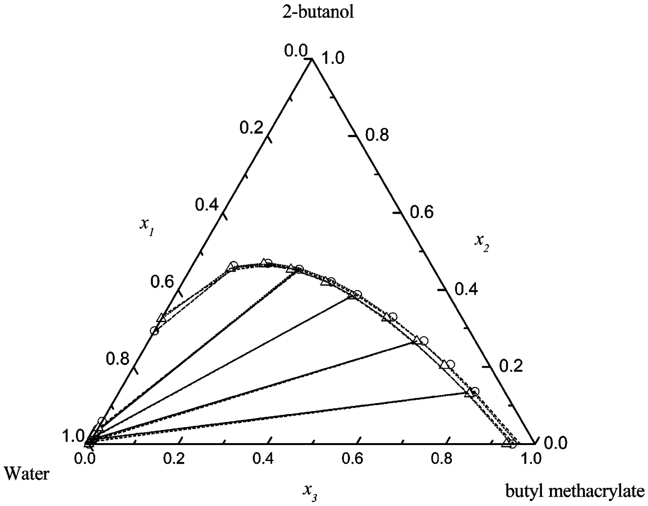

Figure 3. LLE phase diagram for water (1) + 2-butanol (2) + butyl methacrylate (3): ○, exptl data at 288.2 K; Δ, exptl data at 318.2 K; —, exptl tie-line at 288.2 K or 318.2 K; ----, NRTL correlation at 288.2 K or 318.2 K; ····, UNIQUAC correlation at 288.2 K or 318.2 K.

LLE Calculation. The LLE calculation procedure was detailed in Walas.[10] In this paper, the isothermal ternary LLE data were correlated with the NRTL and the UNIQUAC models by adjusting six model parameters simultaneously. The objective function Δ of the parameter determination is defined as

$$\Delta = \left(\sum_{k=1}^{n}\sum_{j=1}^{2}\sum_{i=1}^{3}|(x_{ijk}^{\text{calcd}} - x_{ijk}^{\text{obsd}})|\right)/6n \qquad (2)$$

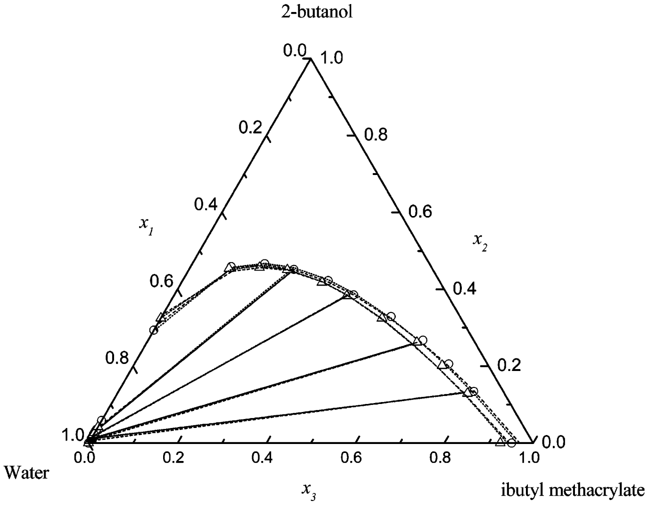

Figure 4. LLE phase diagram for water (1) + 2-butanol (2) + isobutyl methacrylate (3): ○, exptl data at 288.2 K; Δ, exptl data at 318.2 K; —, exptl tie-line at 288.2 K or 318.2 K; ----, NRTL correlation at 288.2 K or 318.2 K; ····, UNIQUAC correlation at 288.2 K or 318.2 K.

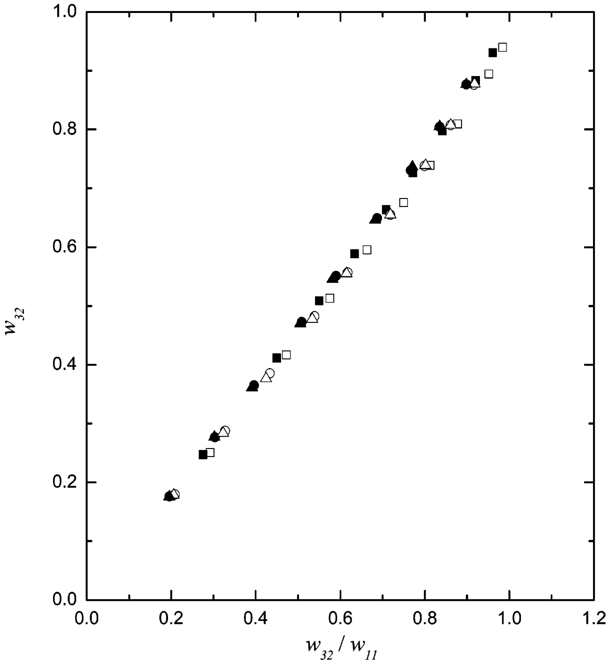

Figure 5. Bachman correlations for three investigated systems at different temperatures: □, methyl methacrylate at 288.2 K; ■, methyl methacrylate at 318.2 K; ○, butyl methacrylate at 288.2 K; ●, butyl methacrylate at 318.2 K; Δ, isobutyl methacrylate at 288.2 K; ▲, isobutyl methacrylate at 318.2 K.

Table 7. Constants of the Bachman Equation System

T/K	A	B	σ
Water (1) + 2-Butanol (2) + Methyl Methacrylate (3)			
288.2	−0.0494	0.9842	0.0123
318.2	−0.0355	0.9941	0.0070
Water (1) + 2-Butanol (2) + Butyl Methacrylate (3)			
288.2	−0.0346	0.9737	0.0104
318.2	−0.0265	0.9921	0.0074
Water (1) + 2-Butanol (2) + Isobutyl Methacrylate (3)			
288.2	−0.0328	0.9714	0.0108
318.2	−0.0257	0.9925	0.0065

Table 8. Correlated Results from the NRTL Model

mixture[a]	T/K	α_{ij}[b]	i−j	a_{ij}[c]/K	a_{ij}[c]/K	Δ[d]
M1	288.2	0.2	1−2	1398.16	−425.74	0.0019
			1−3	1449.35	340.57	
			2−3	−442.57	1177.47	
	318.2	0.2	1−2	1610.87	−472.70	0.0007
			1−3	1622.65	298.61	
			2−3	−526.94	1033.50	
M2	288.2	0.2	1−2	1208.58	−326.73	0.0041
			1−3	1862.50	525.78	
			2−3	731.55	−537.72	
	318.2	0.2	1−2	1469.86	−409.16	0.0038
			1−3	2169.53	490.56	
			2−3	1862.31	−1023.99	
M3	288.2	0.2	1−2	1253.28	−347.06	0.0041
			1−3	1967.12	562.01	
			2−3	779.10	−540.72	
	318.2	0.2	1−2	1463.97	−405.65	0.0041
			1−3	2074.33	445.23	
			2−3	1943.91	−1049.98	

[a] M1: water (1) + 2-butanol (2) + methyl methacrylate (3). M2: water (1) + 2-butanol (2) + butyl methacrylate (3). M3: water (1) + 2-butanol (2) + isobutyl methacrylate (3). [b] α is the nonrandomness parameter in the NRTL model. [c] $a_{ij} = (g_{ij} - g_{jj})/R$ is the parameter of the NRTL model. [d] $\Delta = (\sum_{k=1}^{n} \sum_{j=1}^{2} \sum_{i=1}^{3} |(x_{ijk}^{\text{calcd}} - x_{ijk}^{\text{obsd}})|)/6n$, where n is the number of tie-lines.

Table 9. Correlated Results from the UNIQUAC Model

mixture[a]	T/K	i−j	b_{ij}[b]/K	b_{ji}[b]/K	Δ[c]
M1	288.2	1−2	−176.18	8.52	0.0015
		1−3	−165.35	−506.22	
		2−3	158.62	−381.94	
	318.2	1−2	−280.31	62.70	0.0005
		1−3	−200.30	−476.58	
		2−3	157.95	−311.69	
M2	288.2	1−2	−250.18	67.63	0.0017
		1−3	−634.46	−459.74	
		2−3	157.56	−323.75	
	318.2	1−2	−331.88	102.00	0.0015
		1−3	−833.03	−425.75	
		2−3	181.34	−302.44	
M3	288.2	1−2	−246.01	63.63	0.0016
		1−3	−502.65	−493.55	
		2−3	152.89	−318.98	
	318.2	1−2	−349.07	115.31	0.0021
		1−3	−1024.32	−392.00	
		2−3	184.65	−290.35	

	structural parameters[11]	
component	r	q
water	0.9200	1.400
2-butanol	3.9235	3.664
methyl methacrylate	4.2995	3.884
butyl methacrylate	6.3227	5.504
isobutyl methacrylate	6.3219	5.500

[a] M1: water (1) + 2-butanol (2) + methyl methacrylate (3). M2: water (1) + 2-butanol (2) + butyl methacrylate (3). M3: water (1) + 2-butanol (2) + isobutyl methacrylate (3). [b] $b_{ij} = (u_{ij} - u_{jj})/R$ is the parameter of the UNIQUAC model. [c] $\Delta = (\sum_{k=1}^{n} \sum_{j=1}^{2} \sum_{i=1}^{3} |(x_{ijk}^{\text{calcd}} - x_{ijk}^{\text{obsd}})|)/6n$, where n is the number of tie-lines.

where n is the number of tie-lines and x_{ijk}^{calcd} and x_{ijk}^{obsd} are the calculated and the observed mole fractions of component i in phase j on tie-line k, respectively. Tables 8 and 9 present the correlated results from the NRTL and the UNIQUAC models, respectively. In general, the average deviations from the UNIQUAC model are slightly smaller than those from the NRTL model. The value of overall average deviations from UNIQUAC correlations for the three investigated systems is 0.0015 and from NRTL correlations is 0.0031, respectively. Figures 2 to 4 also compare the calculated phase boundaries and tie-lines from the NRTL and UNIQUAC models with the experimental results. As seen from Figures 2 to 4, good agreements between the calculated results from the solution models and experimental values have been obtained for the three investigated systems.

Conclusions

Liquid−liquid equilibrium (LLE) data for the systems water + 2-butanol + methyl methacrylate, butyl methacrylate, and isobutyl methacrylate were determined at atmospheric pressure over temperatures of (288.2 and 318.2) K. All the investigated systems formed a Type 2 phase diagram of LLE. The area of the two-phase region with butyl methacrylate and isobutyl methacrylate is similar in size, whereas that with methyl methacrylate is appreciably smaller. In general, the phase boundaries and tie-lines of LLE could be correlated well with the NRTL and the UNIQUAC models in this research.

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