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Liquid−Liquid Equilibria for the Ternary System Water + Dodecane + 2-Butyloxyethanol in the Temperature Range from 25 °C to 65 °C

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Liquid−liquid equilibria of the ternary system water + dodecane + 2-butyloxyethanol in the temperature range from 25.0 °C to 65.0 °C were performed at atmospheric pressure. The experimental data were correlated with the UNIQUAC model and good agreement was obtained. For the system with a three-liquid-phase-coexisting region, all six UNIQUAC interaction parameters can be numerically regressed by simply minimizing the deviation of the compositions of three coexisting liquid phases.

Introduction

Nonionic surfactants of the homologous series of polyoxyethylene alcohol $CH_3(CH_2)_{i-1}(OCH_2CH_2)_jOH$, abbreviated by C_iE_j hereafter, are widely used as emulsifying agents and detergents. Precise liquid−liquid equilibrium data of mixtures of the type water + oil + C_iE_j are needed in several industrial applications, for example, herbicides, tertiary oil recovery, and the production of drugs.[1,2] In addition, the phase behavior of mixtures of the type water + oil + C_iE_j also plays an important role in fundamental research of critical phenomena, phase conductivity, and wetting transitions.[3−7] In our laboratory, we have performed liquid−liquid equilibrium measurements on water + C_iE_j binary systems[8] and three ternary systems: water + octane + C_4E_2,[9] water + dodecane + C_6E_2,[10] and water + tetradecane + C_6E_2.[11]

In this study, liquid−liquid equilibrium measurements were performed for the ternary system water + dodecane + 2-butyloxyethanol (C_4E_1) in the temperature range from 25.0 °C to 65.0 °C at atmospheric pressure. Kilpatrick et al.[12] have studied the phase behavior of the ternary mixture water + alkane + C_4E_1 at 25 °C and 30 °C for five different alkanes: hexane, octane, nonane, decane, and tetradecane. On the other hand, Brandani et al.[13] have reported the phase behavior of the ternary mixture water + decane + C_4E_1 at three different temperatures: 25 °C, 30 °C, and 35 °C. To the best of our knowledge, there are no liquid−liquid equilibrium data for the system water + dodecane + C_4E_1 available in the literature. The experimental data were further correlated with a universal quasi-chemical (UNIQUAC) model[14] to estimate six effective binary interaction parameters at different temperatures. The phase behavior of the ternary system was successfully described by the UNIQUAC model.

Experimental Section

Dodecane was obtained from Merck with a purity of 99%. The nonionic surfactant 2-butyloxyethanol was an Aldrich Chemical product with a purity of 99%. These two chemicals were used as received without further purification. Water was purified by a Millipore Milli-RO PLUS 10 and Milli-Q system with a resistivity >18.2 MΩ-cm.

Three samples were prepared in the same mass fraction and placed in a computer-controlled thermostat, whose temperature was controlled within ±0.005 °C, and left to equilibrate for at least 24 h. A quartz thermometer (HP2804a, Hewlett-Packard Co.) was used to detect the temperature, which was directly connected to a personal computer as an input datum to a homemade PID-controlled computer program to adjust the power supply to a heating coil in the thermostat. After equilibrium was reached, each phase was analyzed with a gas chromatograph (China Chromatography 9800, Taiwan) equipped with a thermal conductivity detector. The signal was transferred to an integrator (C-R6A, Chromatopac, Shimadzu Co., Japan) to record area fractions. The temperatures of the injector port and the thermal conductivity detector were held at 270 °C and 300 °C, respectively. The flow rate of the carrier gas, helium, was maintained at 45 $mL\cdot min^{-1}$. The sample was isothermally separated at 235 °C in a 2-m-long × 3.175-mm-diameter stainless steel column packed with Poropak P 80/100 mesh. Each analysis took about 5 min. Single-phase binary mixtures, water + C_4E_1 and dodecane + C_4E_1, with known compositions were used to calibrate the instrument in the composition range of interest. At least three measurements were performed for each phase in every sample. The experimental uncertainty of the gas chromatography was within ±0.0008 mass fraction.

When the water content in the dodecane-rich phase was very low, a Karl Fischer moisture titrator (MKC-210, Kyoto Electronics Co., Japan) was used to further confirm the results of gas chromatography. The experimental uncertainty of the Karl Fischer moisture measurements was within ±0.00002 mass fraction for the water content.

Results and Discussion

The experimental compositions of the equilibrium phases of the ternary system water + dodecane + C_4E_1 at 25 °C, 35 °C, and 45 °C are given in Tables 1, 2, and 3, respectively. According to Kahlweit and Strey,[15] the upper and lower critical solution temperatures for the ternary system water + dodecane + C_4E_1 are ≈33 °C and ≈73 °C, respectively. There is only one two-liquid-phase-coexisting region in the triangle phase diagram of the system at 25 °C, as shown in Figure 1. Note that the surfactant C_4E_1 mainly partitions into the lower aqueous phase and the composition of C_4E_1 is relatively small in the upper oil-

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Table 1. Experimental and Calculated Mass Fractions of Equilibrium Liquid Phases for the Ternary System Water + Dodecane + C_4E_1 at 25.0 °C

experimental data				calculated results			
oil-rich phase		water-rich phase		oil-rich phase		water-rich phase	
H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1
0.0002	0.0263	0.8471	0.1529	0.0003	0.0042	0.9095	0.0898
0.0007	0.0571	0.8824	0.1176	0.0004	0.0071	0.8365	0.1622
0.0008	0.0592	0.7915	0.2060	0.0004	0.0103	0.7538	0.2438
0.0009	0.0603	0.6659	0.3250	0.0005	0.0145	0.6462	0.3485
0.0010	0.0607	0.5773	0.4057	0.0006	0.0193	0.5450	0.4440
0.0011	0.0616	0.5558	0.4255	0.0007	0.0232	0.4807	0.5020
0.0011	0.0624	0.4834	0.4905	0.0008	0.0277	0.4230	0.5511
0.0012	0.0635	0.4230	0.5425	0.0010	0.0346	0.3561	0.6028
0.0013	0.0648	0.3557	0.5974	0.0013	0.0486	0.2690	0.6563
0.0018	0.0717	0.2719	0.6563	0.0018	0.0683	0.1970	0.6801
0.0030	0.0914	0.2042	0.6886	0.0031	0.1160	0.1118	0.6583
0.0033	0.1152	0.0860	0.5971	0.0039	0.1394	0.0891	0.6343
average error: $\sum_{i=1}^{N} \frac{\lvert X_i^{\text{expt}} - X_i^{\text{calc}} \rvert}{N}$				0.0003	0.03	0.05	0.05

N is the number of tie lines

Table 2. Experimental and Calculated Mass Fractions of Equilibrium Liquid Phases for the Ternary System Water + Dodecane + C_4E_1 at 35.0 °C

experiemental data						calculated results					
oil-rich phase		surfactant-rich phase		water-rich phase		oil-rich phase		surfactant-rich phase		water-rich phase	
H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1
Three-Liquid-Phase-Coexisting											
0.0020	0.0849	0.4697	0.4967	0.8471	0.1529	0.0054	0.0668	0.4597	0.5111	0.8313	0.1679
Two-Liquid-Phase-Coexisting Region on the Water/Dodecane Side											
0.0004	0.0265			0.9481	0.0519	0.0016	0.0290			0.9481	0.0517
0.0007	0.0644			0.9018	0.0982	0.0040	0.0546			0.8880	0.1116
Two-Liquid-Phase-Coexisting Region on the C_4E_1/Dodecane Side											
0.0010	0.0816	0.4229	0.5342			0.0056	0.0689	0.4233	0.5391		
0.0020	0.0878	0.3069	0.6253			0.0074	0.0849	0.3047	0.6143		
0.0020	0.0942	0.2562	0.6555			0.0092	0.1006	0.2500	0.6386		
0.0030	0.1187	0.1807	0.6783			0.0136	0.1413	0.1755	0.6568		
0.0060	0.1592	0.1450	0.6665			0.0168	0.1741	0.1415	0.6578		
0.0110	0.2028	0.1206	0.6441			0.0193	0.2021	0.1205	0.6559		
Two-Liquid-Phase-Coexisting Region on the Water/C_4E_1 Side											
		0.5235	0.4552	0.8252	0.1729			0.4767	0.5009	0.8267	0.1725
average error: $\sum_{i=1}^{N} \frac{\lvert X_i^{\text{expt}} - X_i^{\text{calc}} \rvert}{N}$						0.006	0.009	0.007	0.01	0.003	0.003

N is the number of tie lines

Table 3. Experimental and Calculated Mass Fractions of Equilibrium Liquid Phases for the Ternary System Water + Dodecane + C_4E_1 at 45.0 °C

experimental data						calculated results					
oil-rich phase		surfactant-rich phase		water-rich phase		oil-rich phase		surfactant-rich phase		water-rich phase	
H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1
Three-Liquid-Phase-Coexisting											
0.0034	0.1151	0.3356	0.6057	0.8997	0.1003	0.0074	0.0859	0.3536	0.5823	0.8783	0.1212
Two-Liquid-Phase-Coexisting Region on the Water/Dodecane Side											
0.0013	0.0760			0.9261	0.0739	0.0053	0.0675			0.9140	0.0857
0.0002	0.0249			0.9633	0.0367	0.0015	0.0287			0.9640	0.0358
Two-Liquid-Phase-Coexisting Region on the C_4E_1/Dodecane Side											
0.0037	0.1205	0.2583	0.6493			0.0102	0.1086	0.2495	0.631		
0.0045	0.1364	0.2083	0.6679			0.0131	0.1338	0.1969	0.6422		
0.0099	0.1877	0.1535	0.6628			0.0189	0.1866	0.1382	0.6387		
0.0160	0.2329	0.1253	0.6318			0.0221	0.219	0.1161	0.6314		
Two-Liquid-Phase-Coexisting Region on the Water/C_4E_1 Side											
		0.3685	0.5923	0.8954	0.1046			0.3888	0.5728	0.8802	0.1194
average error: $\sum_{i=1}^{N} \frac{\lvert X_i^{\text{expt}} - X_i^{\text{calc}} \rvert}{N}$						0.006	0.009	0.006	0.009	0.005	0.005

N is the number of tie lines

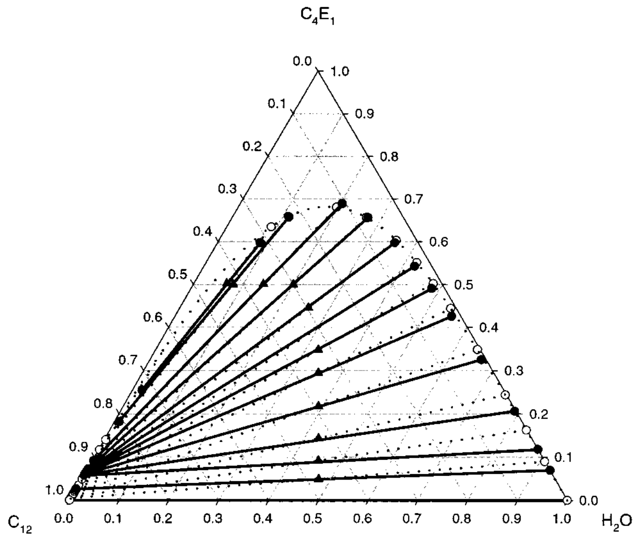

Figure 1. Ternary liquid−liquid equilibria (mass fraction) for the system water + dodecane + C_4E_1 at 25.0 °C: experimental tie lines (●, solid line); calculated binodal curve (dotted curve) and tie lines (○, dotted line); total compositions (▲).

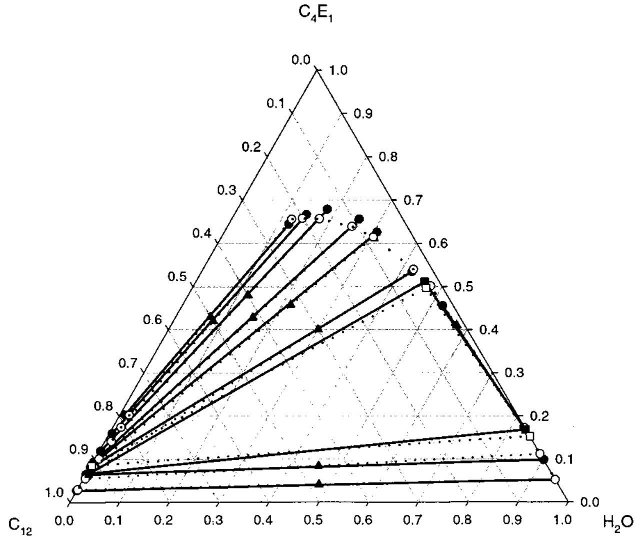

Figure 2. Ternary liquid−liquid equilibria (mass fraction) for the system water + dodecane +C_4E_1 at 35.0 °C: experimental tie lines (●, solid line); calculated binodal curves (dotted curve) and tie lines (○, dotted line); total compositions (▲); experimental three-liquid-phase-coexisting tie triangle (■, solid line); calculated three-liquid-phase-coexisting tie triangle (□, dotted line).

rich phase. The fitting coefficient of a linear expression to each tie line and its corresponding total composition data point is always better than 0.999.

When the temperature is increased above the lower critical solution temperature, ≈33 °C, the system should have a three-liquid-phase-coexisting region. As expected, the system at 35 °C and 45 °C exhibits one three-liquid-phase-coexisting tie triangle region and three two-liquid-phase-coexisting envelopes, as shown in Figures 2 and 3.

In the interval of the upper and lower critical solution temperatures, the effect of temperature on the composition of the three-liquid-phase-coexisting region was examined. Table 4 lists the experimental results of the equilibrium compositions of the three-liquid-phase-coexisting tie triangle in every 5 °C from 35 °C to 65 °C. It is obvious that the apex of the three-liquid-phase-coexisting tie triangle, that is, the C_4E_1-rich phase, shifts counterclockwise with an increase in temperature, as shown in Figure 4, consistent with the observation of Kahlweit and Strey.[15] Note that the surfactant moves continuously from the water-rich phase to the oil-rich phase along with increasing temperature, as shown in Table 4.

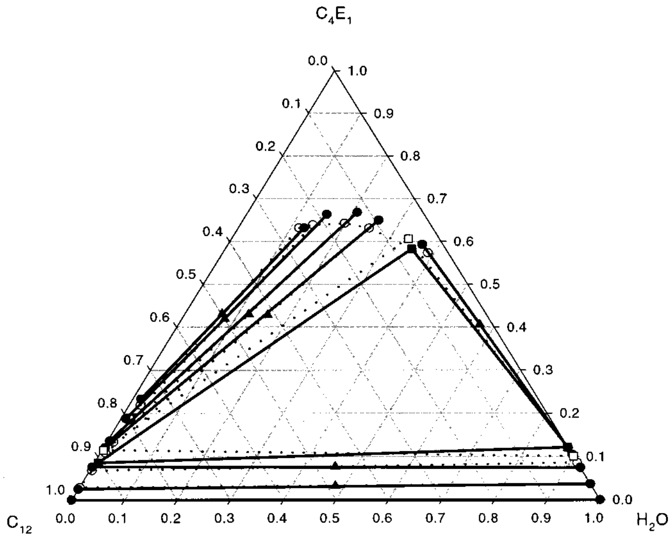

Figure 3. Ternary liquid−liquid equilibria (mass fraction) for the system water + dodecane + C_4E_1 at 45.0 °C: experimental tie lines (●, solid line); calculated binodal curves (dotted curve) and tie line (○, dotted line); total compositions (▲); experimental three-liquid-phase-coexisting tie triangle (▲, solid line); calculated three-liquid-phase-coexisting tie triangle (□, dotted line).

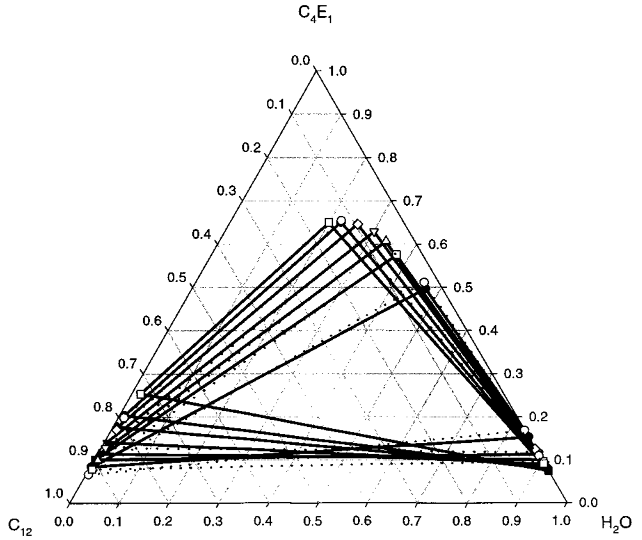

Figure 4. Variation of the liquid−liquid equilibria (mass fraction) of three-liquid-phase-coexisting tie triangle for the system water + dodecane +C_4E_1 as a function of temperature ranging from 35.0 °C to 65.0 °C: experimental three-liquid-phase-coexisting tie triangle [(●) 35.0 °C; (■) 40.0 °C; (▲) 45.0 °C; (▼) 50.0 °C; (♦) 55.0 °C; (●) 60.0 °C; (■) 65.0 °C; solid symbol and solid line]. Calculated three-liquid-phase-coexisting tie triangle [(○) 35.0 °C; (□) 40.0 °C; (△) 45.0 °C; (▽) 50.0 °C; (◊) 55.0 °C; (○) 60.0 °C; (□) 65.0 °C; open symbol and dotted line].

The UNIQUAC model of Abrams and Prausnitz[14] was used to correlate the experimental data. In this study, the relative van der Waals volume r_i and van der Waals surface area q_i were adopted from the UNIFAC group contribution of Hansen et al.,[16] listed in Table 5. The effective binary interaction parameter, a_{ij}, is defined by

$$a_{ij} = \frac{u_{ij} - u_{jj}}{R} \tag{1}$$

Table 4. Experimental Mass Fractions of Equilibrium Liquid Phases of a Three-Liquid-Phase-Coexisting Tie Triangle for the Ternary System Water + Dodecane + C_4E_1 from 35.0 °C to 65.0 °C

	experimental data						calculated results					
	dodecane -rich phase		C_4E_1-rich phase		water-rich phase		dodecane -rich phase		C_4E_1-rich phase		water-rich phase	
t (°C)	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1	H_2O	C_4E_1
35	0.0021	0.0849	0.4697	0.4967	0.8471	0.1529	0.0054	0.0668	0.4597	0.5111	0.8313	0.1679
40	0.0026	0.1013	0.373	0.5738	0.8873	0.1127	0.0067	0.0789	0.3721	0.5759	0.9033	0.0964
45	0.0034	0.1151	0.3356	0.6057	0.8997	0.1003	0.0074	0.0999	0.3352	0.6078	0.8885	0.1112
50	0.0055	0.1417	0.3008	0.6288	0.9102	0.0898	0.0084	0.1290	0.3004	0.6293	0.8821	0.1172
55	0.0091	0.1771	0.2596	0.6459	0.9170	0.0830	0.0102	0.1709	0.2597	0.6459	0.8738	0.1247
60	0.0105	0.2037	0.2238	0.6504	0.9221	0.0779	0.0120	0.1983	0.2224	0.6543	0.8891	0.1088
65	0.0179	0.2543	0.1995	0.6496	0.9259	0.0741	0.0182	0.2530	0.1990	0.6506	0.9083	0.0905
average error: $\sum_{i=1}^{N} \frac{\lvert X_i^{\text{expt}} - X_i^{\text{calc}} \rvert}{N}$							0.003	0.01	0.002	0.003	0.02	0.02

N is the number of data points

Table 5. The Relative van der Waals Volume *r* and van der Waals Surface Area *q*[16]

compound	*r*	*q*
water	0.9200	1.4000
2-butyloxyethanol	5.0558	4.3720
dodecane	8.5462	7.0600

where *R* is the gas constant and u_{ij} is the UNIQUAC interaction parameter between molecules *i* and *j*.

There are two effective binary interaction parameters for a pair of substances. Therefore, six effective binary interaction parameters are required for a ternary system. For the system at 25.0 °C, there is only one two-liquid-phase-coexisting region. These six effective binary interaction parameters were determined by numerically minimizing the following objective function:[17]

$$F_x = \sum_{k=1}^{12} \sum_{j=1}^{2} \sum_{i=1}^{3} \left(\frac{x_{ijk}^{\text{expl}} - x_{ijk}^{\text{calc}}}{x_{ijk}^{\text{expl}}} \right)^2 \tag{2}$$

where x_{ijk}^{expl} and x_{ijk}^{calc} are the experimental and calculated, respectively, mole fraction of component *i* in phase *j* along a tie line *k*. The liquid−liquid equilibrium flash calculation[18] was applied to evaluate the compositions of both liquid phases along each tie line by using the experimental total compositions as input data. As one can see in Figure 1, the predicted binodal curve was in very good agreement with the experimental result.

On the other hand, for the systems at 35.0 °C and higher temperatures, there are one three-liquid-phase-coexisting tie triangle region and three two-liquid-phase-coexisting envelopes in each triangle phase diagram. Simply the compositions of the three-liquid-phase-coexisting tie triangle were used to determine the six effective binary interaction parameters for the systems at 35.0 °C and higher temperatures by numerically minimizing the following objective function:[17,19,20]

$$F_x = \sum_{j=1}^{3} \sum_{i=1}^{3} \left(\frac{x_{ij}^{\text{expl}} - x_{ij}^{\text{calc}}}{x_{ij}^{\text{expl}}} \right)^2 \tag{3}$$

where x_{ij}^{expl} and x_{ij}^{calc} are the experimental and calculated, respectively, mole fraction of component *i* in phase *j* of a three-liquid-phase-coexisting tie triangle. It should be pointed out that the experimental results of three two-liquid-phase-coexisting regions were not used to correlate the interaction parameters. Once all the six effective binary interaction parameters were determined, the liquid−liquid equilibrium flash calculation[18] was applied to directly predict the compositions of both liquid phases along each tie line in all three two-liquid-phase-coexisting envelopes by using the experimental total compositions as input data. As one can see in Figures 2 and 3, the predicted binodal curves were consistent with the experimental results.

Table 6. Effective UNIQUAC Interaction Parameters for the System Water (1) + C_4E_1 (2) + Dodecane (3)

t (°C)	a_{12}	a_{21}	a_{13}	a_{31}	a_{23}	a_{32}
25.0	268.7	−169.1	170.7	2399	−62.25	175.7
35.0	386.2	−193.3	124.9	2338	−38.06	160.9
40.0	364.4	−175.4	103.5	2279	106.8	27.00
45.0	326.0	−145.8	97.38	2245	338.6	−84.58
50.0	192.6	−32.56	73.75	2087	1335	−218.7
55.0	86.15	89.77	26.59	1981	2009	−240.0
60.0	81.33	124.8	11.42	1948	2023	−245.4
65.0	77.35	145.8	1.148	1930	2089	−265.1

The minimization of the objective function F_x, eqs 2 and 3, was accomplished by using the subroutine DUMPOL of the IMSL library.[21] The regression results of the UNIQUAC effective binary interaction parameters are shown in Table 6. Note that these parameters are obviously temperature-dependent. The calculated results for each tie line are given in Tables 1−4 to make a comparison with experimental data. The average absolute deviations between experimental data and calculated results are small and also listed in the bottom of Tables 1−4. The calculated binodal curves are also presented by long dashed curves in Figures 1−3. As one can see in Figures 1−4, the phase behavior of the system water + dodecane + C_4E_1 is successfully described by the UNIQUAC model.

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