

Liquid–Liquid Equilibria of Alkane (C10–C14) + *sec*-Butylbenzene + Sulfolane

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ABSTRACT: Equilibrium tie line data have been determined at (323.15, 348.15, and 373.15) K for the ternary liquid–liquid equilibria (LLE) of three alkane + *sec*-butylbenzene + sulfolane systems, where the alkanes studied are decane, dodecane, and tetradecane. The relative mutual solubility of *sec*-butylbenzene is higher in decane + sulfolane than in dodecane + sulfolane or tetradecane + sulfolane mixtures. The tie line data were correlated with the UNIQUAC and NRTL models. The calculated values based on the UNIQUAC model were found to be better than those based on the NRTL model; the average root-mean-square deviation between the phase composition obtained from experiment and that from calculation was 0.00385 for UNIQUAC compared to 0.00566 for NRTL. The values of selectivity and distribution coefficients were derived from the equilibrium data.

INTRODUCTION

The design and evaluation of industrial units for separation processes requires reliable phase equilibria data of the different mixtures involved in a given process. Especially, ternary liquid–liquid equilibrium (LLE) data are essential for a proper understanding of the solvent extraction process. Sulfolane is used widely in the chemical industry for the extraction of aromatic hydrocarbons,^{1–4} and many investigators^{5–15} have studied liquid–liquid equilibria for the ternary systems containing sulfolane + alkanes (C5–C8) + (benzene, toluene, or xylene) near room temperature, but quantitative phase equilibrium data for the systems containing sulfolane and alkanes with carbon number larger than nine^{16–22} were seldom reported.

Therefore, the purpose of this study was to obtain LLE data for the ternary mixtures decane + *sec*-butylbenzene + sulfolane, dodecane + *sec*-butylbenzene + sulfolane, and tetradecane + *sec*-butylbenzene + sulfolane at (323.15, 348.15, and 373.15) K. The LLE data for these ternary systems were then correlated by the UNIQUAC model of Abrams and Prausnitz²³ and the NRTL model of Renon and Prausnitz.²⁴ The effects of temperature on the selectivity and the distribution coefficient were also discussed.

EXPERIMENTAL SECTION

Chemicals. Sulfolane, decane, dodecane, anisole, and carbon disulfide were supplied by E. Merck with a stated purity of 99.0%. Tetradecane was obtained from Tokyo Kasei Organic Chemical Industry Company with a purity of more than 99.0%. *sec*-Butylbenzene was obtained from the Acros Organics with a purity of more than 99.0%. The purities of all materials were checked by gas chromatograph and found to be better than 99.0%. All compounds were stored in a desiccator with a drying agent before use. They were used without further purification.

Apparatus and Procedure. The experimental tie line data in the two-phase region was determined by using a 50 cm³ jacketed glass cell controlled at a temperature of (323.15, 348.15, or 373.15) K \pm 0.03 K. Specific amounts of alkane, sulfolane, and *sec*-butylbenzene were introduced into the cell and agitated while maintained at a constant temperature by circulation of silicon oil through the external jacket from a thermostatic bath. The sample was stirred for 1 h with a

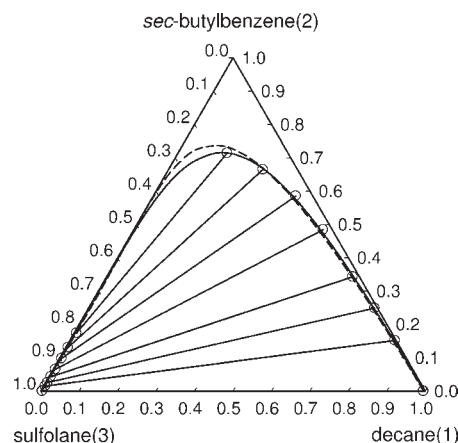


Figure 1. LLE data for the decane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 323.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.

stirrer at a speed of 1100 rpm and then allowed to settle for at least 5 h. After equilibrium was attained, the phases were allowed to separate and small samples of approximately 1 g were taken from each phase. A fixed amount of anisole was added as the internal standard and diluted with 50 g of carbon disulfide⁶ to prepare them for analysis. The accuracy of weighing was \pm 0.0001 g. The sample analysis was performed by using a Hewlett-Packard model 6890 gas chromatograph equipped with a flame ionization detector and a HP Ultra 1 column (cross-linked methyl silicone gum, $25 \text{ m} \times 3.2 \times 10^{-4} \text{ m} \times 5.2 \times 10^{-7} \text{ m}$ film thickness). The injector and detector temperatures were maintained at 573.15 K. The column temperature was programmed for an initial temperature of 353.15 K, maintained for 1 min, and a final temperature of 393.15 K, maintained for 1 min. The heating rate was $5 \text{ K} \cdot \text{min}^{-1}$, and the flow rate of nitrogen carrier gas was $2.5 \times 10^{-5} \text{ m}^3 \cdot \text{min}^{-1}$. The estimated accuracy for the mass

Received: February 28, 2011

Accepted: June 8, 2011

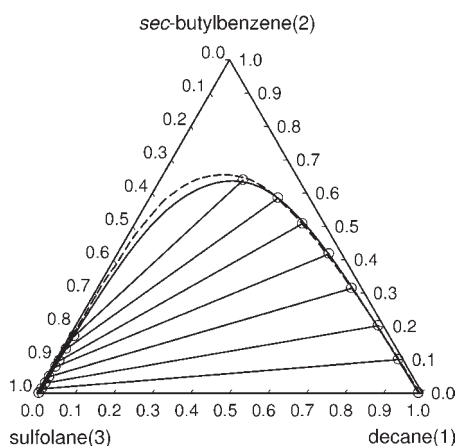
Published: June 23, 2011

Table 1. Experimental LLE Data for the System Decane (1) + *sec*-Butylbenzene (2) + Sulfolane (3)

T	alkane-rich phase			sulfolane-rich phase		
	K	x_{11}	x_{21}	x_{31}	x_{12}	x_{22}
323.15	0.995	0.000	0.005	0.004	0.000	0.996
	0.845	0.152	0.003	0.004	0.015	0.981
	0.744	0.249	0.007	0.005	0.026	0.969
	0.640	0.343	0.018	0.005	0.043	0.952
	0.492	0.484	0.024	0.006	0.066	0.929
	0.371	0.586	0.044	0.006	0.098	0.896
	0.245	0.665	0.090	0.006	0.131	0.863
	0.128	0.714	0.158	0.007	0.174	0.820
	348.15	0.991	0.000	0.009	0.006	0.000
0.888		0.100	0.011	0.007	0.013	0.980
0.785		0.202	0.013	0.008	0.030	0.962
0.660		0.314	0.026	0.008	0.049	0.942
0.550		0.418	0.032	0.010	0.079	0.911
0.435		0.508	0.056	0.011	0.097	0.893
0.334		0.585	0.081	0.011	0.133	0.857
0.216		0.639	0.145	0.012	0.171	0.817
373.15		0.983	0.000	0.018	0.010	0.000
	0.897	0.081	0.021	0.010	0.014	0.976
	0.802	0.167	0.032	0.012	0.029	0.960
	0.718	0.247	0.035	0.011	0.043	0.946
	0.603	0.346	0.052	0.011	0.065	0.923
	0.456	0.457	0.087	0.011	0.105	0.884
	0.308	0.544	0.149	0.013	0.159	0.828
	0.171	0.549	0.280	0.013	0.190	0.797

Table 2. Experimental LLE Data for the System Dodecane (1) + *sec*-Butylbenzene (2) + Sulfolane (3)

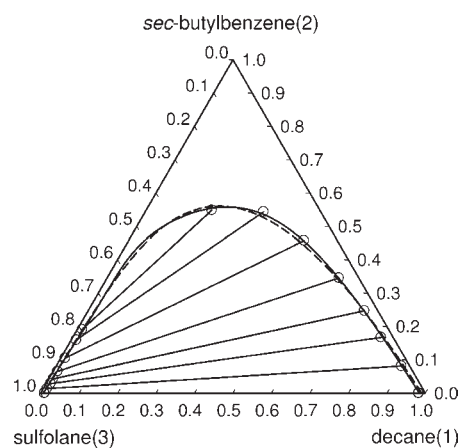
T	alkane-rich phase			sulfolane-rich phase		
	K	x_{11}	x_{21}	x_{31}	x_{12}	x_{22}
323.15	0.994	0.000	0.006	0.002	0.000	0.998
	0.870	0.122	0.008	0.002	0.011	0.987
	0.711	0.277	0.012	0.002	0.028	0.970
	0.576	0.407	0.018	0.003	0.054	0.943
	0.463	0.511	0.026	0.003	0.072	0.925
	0.381	0.580	0.039	0.003	0.089	0.908
	0.292	0.650	0.057	0.004	0.109	0.888
	0.180	0.705	0.114	0.004	0.149	0.847
	348.15	0.989	0.000	0.011	0.004	0.000
0.846		0.141	0.012	0.004	0.014	0.982
0.681		0.295	0.024	0.005	0.042	0.953
0.540		0.427	0.033	0.006	0.074	0.920
0.440		0.512	0.048	0.006	0.096	0.898
0.341		0.586	0.073	0.007	0.122	0.871
0.252		0.651	0.097	0.007	0.159	0.834
0.172		0.681	0.148	0.008	0.202	0.790
373.15		0.979	0.000	0.021	0.005	0.000
	0.839	0.139	0.022	0.009	0.022	0.969
	0.686	0.280	0.034	0.010	0.050	0.940
	0.535	0.408	0.057	0.009	0.077	0.914
	0.385	0.520	0.095	0.009	0.114	0.877
	0.303	0.574	0.124	0.009	0.135	0.856
	0.235	0.603	0.163	0.010	0.165	0.825
	0.157	0.615	0.228	0.011	0.200	0.789

**Figure 2.** LLE data for the decane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 348.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.

fraction of each compound is ± 0.005 . The greatest error in the material balance in these experiments was found to be less than 2%.

RESULTS

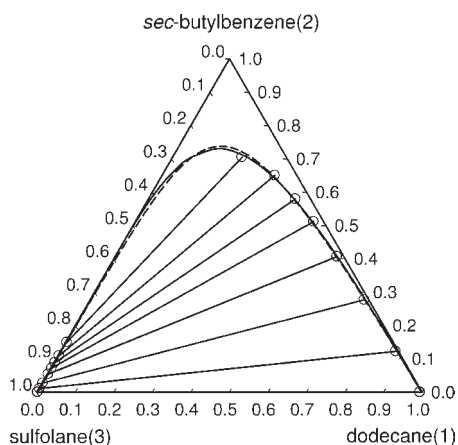
The experimental tie line data are given in Tables 1 to 3 for the ternary systems formed by sulfolane (3) + *sec*-butylbenzene (2) with decane, dodecane, or tetradecane (1), respectively. Concentrations of components i ($i = 1, 2, 3$) in phase L ($L = 1$, alkane-rich phase;

**Figure 3.** LLE data for the decane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 373.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.

$L = 2$, sulfolane-rich phase) are given in mole fraction, x_{iL} . The effect of temperature on the equilibrium for the system decane (1) + *sec*-butylbenzene (2) + sulfolane (3) system is shown in Figure 1. As expected, the size of the two-phase region decreases with an increase in temperature. Similar behavior was also noticed in the other ternary systems. The three systems studied present a wide two-phase region that is important, together with the slope of the tie lines, when establishing the selectivity of sulfolane.

Table 3. Experimental LLE Data for the System Tetradecane (1) + *sec*-Butylbenzene (2) + Sulfolane (3)

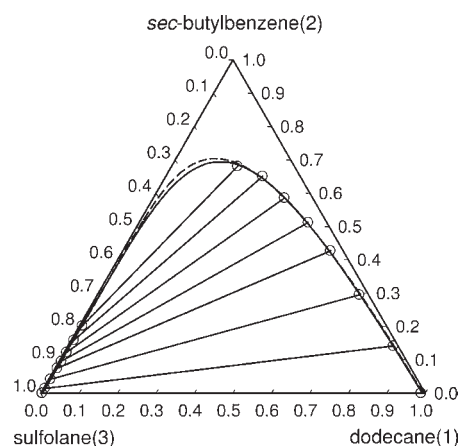
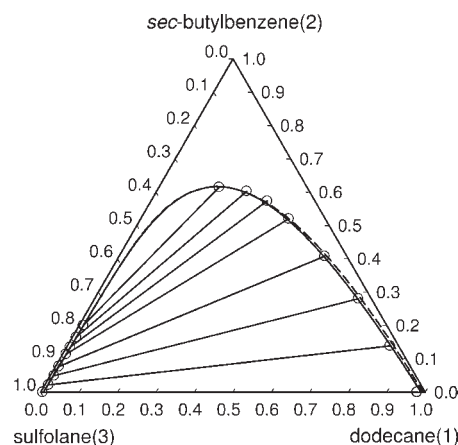
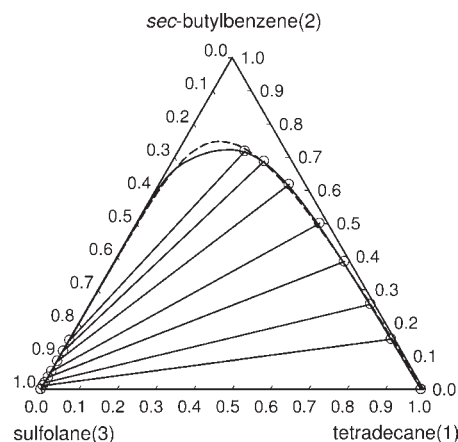
T K	alkane-rich phase			sulfolane-rich phase		
	x_{11}	x_{21}	x_{31}	x_{12}	x_{22}	x_{32}
323.15	0.993	0.000	0.007	0.001	0.000	0.999
	0.840	0.151	0.009	0.001	0.011	0.988
	0.734	0.256	0.010	0.001	0.020	0.979
	0.601	0.385	0.014	0.002	0.037	0.962
	0.478	0.500	0.021	0.002	0.054	0.944
	0.340	0.618	0.042	0.002	0.085	0.913
	0.240	0.688	0.072	0.002	0.115	0.883
	0.175	0.718	0.107	0.002	0.148	0.850
348.15	0.988	0.000	0.012	0.002	0.000	0.998
	0.872	0.113	0.015	0.002	0.009	0.989
	0.740	0.237	0.023	0.003	0.025	0.973
	0.635	0.338	0.027	0.003	0.036	0.962
	0.496	0.466	0.038	0.003	0.065	0.932
	0.360	0.580	0.060	0.004	0.108	0.889
	0.249	0.655	0.096	0.005	0.148	0.847
	0.161	0.676	0.162	0.006	0.197	0.797
373.15	0.977	0.000	0.023	0.004	0.000	0.996
	0.830	0.144	0.027	0.004	0.021	0.975
	0.730	0.236	0.034	0.005	0.037	0.958
	0.630	0.325	0.044	0.005	0.056	0.939
	0.488	0.449	0.063	0.006	0.086	0.908
	0.355	0.545	0.100	0.008	0.125	0.867
	0.237	0.616	0.147	0.007	0.170	0.823
	0.136	0.621	0.242	0.007	0.223	0.770

**Figure 4.** LLE data for the dodecane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 323.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.

Data Correlation. The UNIQUAC and NRTL models were used to correlate and predict the LLE data in the present work. The corresponding sets of binary interaction parameters were determined by minimizing the differences between the experimental and calculated concentrations over all of the tie lines for each ternary system using the Simplex Search method. The optimization was done without any weighting and all data points have the same importance. The objective function (F) used is

$$F = \min \sum_i \sum_L \sum_k (x_{iLk} - x_{iLk}^c)^2 \quad (1)$$

where x and x^c are the experimental and calculated mole fractions, respectively, and the subscripts i , L , and k denote the number of components, phases, and tie lines, respectively.

**Figure 5.** LLE data for the dodecane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 348.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.**Figure 6.** LLE data for the dodecane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 373.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.**Figure 7.** LLE data for the tetradecane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 323.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.

For the NRTL model, the third nonrandomness parameter, α_{ij} , was set at a value of 0.2.⁸ Perhaps this α_{ij} value is not optimal,

Table 4. UNIQUAC and NRTL Parameters for the Decane (1) + *sec*-Butylbenzene (2) + Sulfolane (3) System at (323.15, 348.15, and 373.15) K As Well As the Calculated Root-Mean-Square Deviation (rmsd)

T		UNIQUAC parameters/K			NRTL parameters/K		
K	i-j	$(u_{ij} - u_{ji})/R$	$(u_{ji} - u_{ii})/R$	rmsd	$(g_{ij} - g_{ji})/R$	$(g_{ji} - g_{ii})/R$	rmsd
323.15	1-2	-76.65	69.14	0.00358	61.89	-275.38	0.01124
	1-3	564.02	72.38		1203.30	1270.90	
	2-3	153.03	36.39		461.58	276.96	
348.15	1-2	-492.75	750.13	0.01115	564.95	-493.48	0.00445
	1-3	514.34	16.48		1374.60	1110.50	
	2-3	325.88	-155.38		470.92	321.46	
373.15	1-2	-290.54	555.86	0.00286	-244.28	441.85	0.00595
	1-3	461.13	55.54		1133.60	1281.10	
	2-3	204.80	18.57		29.45	745.03	

Table 5. UNIQUAC and NRTL Parameters for the Dodecane (1) + *sec*-Butylbenzene (2) + Sulfolane (3) System at (323.15, 348.15, and 373.15) K As Well As the Calculated Root-Mean-Square Deviation (rmsd)

T		UNIQUAC parameters/K			NRTL parameters/K		
K	i-j	$(u_{ij} - u_{ji})/R$	$(u_{ji} - u_{ii})/R$	rmsd	$(g_{ij} - g_{ji})/R$	$(g_{ji} - g_{ii})/R$	rmsd
323.15	1-2	-98.63	96.31	0.00252	-808.70	585.52	0.00921
	1-3	654.15	28.45		1480.40	1248.40	
	2-3	159.90	34.31		306.02	322.10	
348.15	1-2	9.76	-21.75	0.00231	110.99	-193.65	0.00266
	1-3	564.32	-0.44		1226.10	1383.00	
	2-3	193.36	9.45		424.20	344.19	
373.15	1-2	235.99	-171.50	0.00270	262.46	-211.16	0.00364
	1-3	575.14	-12.94		1356.70	1230.40	
	2-3	152.74	47.00		219.45	568.09	

Table 6. UNIQUAC and NRTL Parameters for the Tetradecane (1) + *sec*-Butylbenzene (2) + Sulfolane (3) System at (323.15, 348.15, and 373.15) K As Well As the Calculated Root-Mean-Square Deviation (rmsd)

T		UNIQUAC parameters/K			NRTL parameters/K		
K	i-j	$(u_{ij} - u_{ji})/R$	$(u_{ji} - u_{ii})/R$	rmsd	$(g_{ij} - g_{ji})/R$	$(g_{ji} - g_{ii})/R$	rmsd
323.15	1-2	-389.47	721.59	0.00485	-524.37	-194.69	0.00627
	1-3	492.78	47.29		1355.50	1300.70	
	2-3	275.29	-51.55		344.34	353.59	
348.15	1-2	-348.62	685.35	0.00220	-184.31	-97.94	0.00432
	1-3	470.73	59.51		1358.00	1492.60	
	2-3	253.10	-26.83		290.36	431.81	
373.15	1-2	-303.50	551.13	0.00251	-342.53	422.47	0.00323
	1-3	491.84	4.36		1035.20	1449.10	
	2-3	285.51	-33.87		177.34	589.76	

but the NRTL model generally correlates fairly well with the LLE data at $\alpha_{ij} = 0.2$ in relative to other α_{ij} values. The parameters calculated are shown in Tables 4 to 6, together with the root-mean-square deviation (rmsd) values, defined as:

$$\text{rmsd} = \left[\sum_i \sum_L \sum_k (x_{iLk} - x_{iLk}^c)^2 / 6N \right]^{1/2} \quad (2)$$

where N = number of tie lines.

As can be seen from Tables 4 to 6, the calculation based on both the UNIQUAC and the NRTL models gave good representation of

the tie-line data for these systems. However, the UNIQUAC model, fitted to the experimental data, is more accurate than the NRTL model, according to the analysis of rmsd (the average root-mean-square deviation phase composition error was 0.00385 for UNIQUAC as compared to 0.00566 for NRTL).

DISCUSSION

The slopes of the tie lines presented in Figures 2 to 7 show that *sec*-butylbenzene is more soluble in alkanes than in sulfolane. It is observed that the relative mutual solubility of *sec*-butylbenzene is

Table 7. Experimental Selectivity Values S for Each Tie Line

S for the system	323.15 K	348.15 K	373.15 K
decane	18.7	15.7	14.6
	15.7	14.9	11.6
	15.4	12.5	11.0
	11.9	10.5	10.0
	10.7	7.8	9.4
	7.9	7.0	7.1
dodecane	4.7	4.8	4.6
	36.2	20.9	14.2
	30.7	19.8	12.1
	29.6	16.2	11.1
	23.3	13.2	9.2
	18.8	10.9	7.6
tetradecane	14.0	8.8	6.4
	10.6	6.7	4.7
	51.9	32.6	28.9
	41.1	31.0	25.1
	38.0	27.0	21.0
	27.2	21.7	16.1
	23.4	17.6	10.9
	19.1	11.7	9.0
	15.0	8.0	6.8

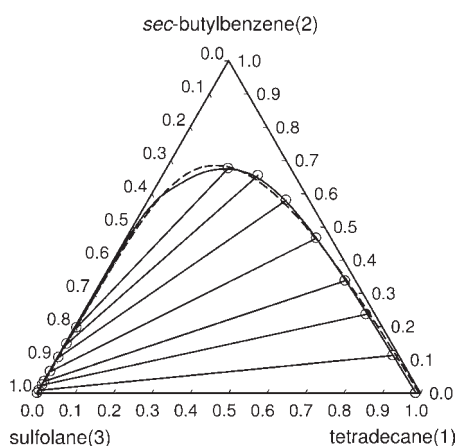


Figure 8. LLE data for the tetradecane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 348.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.

higher in decane + sulfolane than in dodecane + sulfolane or tetradecane + sulfolane mixtures at the same temperature. This solubility effect is reflected in the size of the two-phase region, increasing slightly in the order tetradecane > dodecane > decane at the same temperature.

The effectiveness of extraction of an aromatic compound by sulfolane is given by its selectivity (S), which is a measure of the ability of sulfolane to separate aromatics from alkanes

$$S = (x_2/x_1)_{\text{sulfolane-rich phase}} / (x_2/x_1)_{\text{alkane-rich phase}} \quad (3)$$

where subscript 2 represents *sec*-butylbenzene and subscript 1 represents decane, dodecane, or tetradecane.

This quantity is not constant over the whole two-phase region. Table 7 lists experimental values of S . From the data, we find the

Table 8. Experimental Distribution Coefficient Values κ for Each Tie Line

κ for the system	323.15 K	348.15 K	373.15 K
decane	0.097	0.128	0.166
	0.103	0.149	0.171
	0.125	0.157	0.173
	0.136	0.189	0.189
	0.167	0.190	0.230
	0.197	0.226	0.293
dodecane	0.243	0.267	0.347
	0.092	0.101	0.159
	0.099	0.143	0.178
	0.133	0.174	0.190
	0.141	0.187	0.219
	0.153	0.208	0.235
tetradecane	0.167	0.244	0.273
	0.212	0.297	0.326
	0.074	0.079	0.146
	0.078	0.105	0.158
	0.095	0.106	0.173
	0.108	0.140	0.191
	0.138	0.186	0.230
	0.167	0.226	0.276
	0.206	0.291	0.359

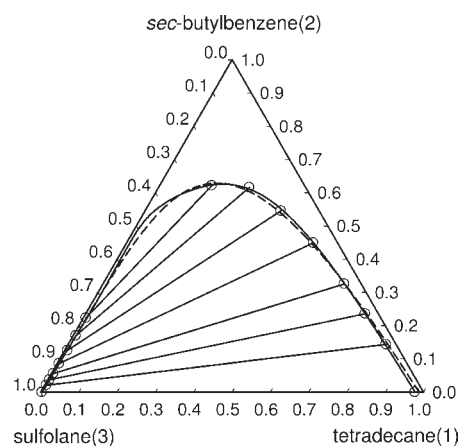


Figure 9. LLE data for the tetradecane (1) + *sec*-butylbenzene (2) + sulfolane (3) system at 373.15 K. Curves calculated by —, UNIQUAC model; - - -, NRTL model; ○—○, experimental tie line.

tendency that for the same system, the higher the temperature, the lower the selectivity. At the same temperature for the different system, the order of the selectivity of sulfolane to *sec*-butylbenzene is tetradecane > dodecane > decane. Since the selectivity in all cases is greater than one, it means that extraction is possible.

The distribution coefficient of *sec*-butylbenzene over the two liquid phases in equilibrium is defined as

$$\kappa = (x_2)_{\text{sulfolane-rich phase}} / (x_2)_{\text{alkane-rich phase}} \quad (4)$$

This coefficient is related to the number of theoretical stages that are necessary for a given extraction. The lower the number of theoretical stages is necessary for the larger κ values. The

experimental values of κ in this study are presented in Table 8. We can find the tendency that for the same system, the higher the temperature, the larger the κ value. Also in our three different systems, there are not obvious differences between the κ values at the same temperature.

Comparing with our previous systems^{16,17} of alkane (C10–C14) + *n*-butylbenzene + sulfolane, the results seem to similar based on temperature and carbon-number trends; however, slightly higher selectivity and distribution coefficient values were observed for the *n*-butylbenzene system (in previous work) at the same temperature. There exists difference that sulfolane is more selective to *n*-butylbenzene than to *sec*-butylbenzene (in this work). This is because the different steric effects present in the isomers between *n*-butylbenzene and *sec*-butylbenzene.

CONCLUSIONS

Liquid–liquid equilibrium data of the ternary systems decane (1) + *sec*-butylbenzene (2) + sulfolane (3), dodecane (1) + *sec*-butylbenzene (2) + sulfolane (3), and tetradecane (1) + *sec*-butylbenzene (2) + sulfolane (3) were determined at (323.15, 348.15, and 373.15) K, respectively.

From the selectivity data, the separation of *sec*-butylbenzene from decane, dodecane, or tetradecane by extraction with sulfolane is feasible. The calculation based on the UNIQUAC and NRTL models showed that the best results are given by the UNIQUAC model. The binodal curves calculated by the UNIQUAC model for the systems studied here were drawn to investigate the temperature dependence. The size of the two-phase region decreases with increasing temperature.

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Funding Sources

The authors thank the National Science Council, NSC 99-2622-E-168-011-CC3, for the financial support of this work.

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