Influence of the Temperature on the Liquid–Liquid Equilibria of Heptane + Toluene + Sulfolane and Heptane + *m*-Xylene + Sulfolane

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Equilibrium tie line data have been determined at (298.15, 323.15, 348.15, and 373.15) K for the ternary liquid–liquid equilibria (LLE) of heptane + (toluene or *m*-xylene) + sulfolane systems. The relative mutual solubility of toluene is higher than that of *m*-xylene in heptane + 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 0.5111 for UNIQUAC compared to 0.6475 for NRTL. The values of selectivity and the distribution coefficient were derived from the equilibrium data at different temperatures.

Introduction

Accurate phase equilibrium data are important parameters for the design and evaluation of industrial unit operations for the extraction processes. Especially, ternary liquid–liquid equilibria (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[5–16] 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 + heptane + (toluene or *m*-xylene) at higher temperatures are scarce.

Therefore, the purpose of this study was to obtain LLE data for the ternary mixtures heptane + toluene + sulfolane and heptane + *m*-xylene + sulfolane at (298.15, 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 Prausnitz[17] and the NRTL model of Renon and Prausnitz.[18] The effects of temperature on the selectivity and the distribution coefficient were also discussed.

Experimental Section

Chemicals. The supplier, grade, and purity of each substance used are given in Table 1. All compounds were stored in a desiccator with a drying agent before use. They were used without further purification.

Table 1. Supplier, Grade, and Purity of Materials Used

substance	supplier	grade	purity %[a]
sulfolane[b]	Merck	for synthesis	>99.0
heptane	Merck	GR	>99.0
toluene	Merck	GR	>99.5
m-xylene	Merck	for synthesis	>99.0
anisole	Merck	for synthesis	>99.0
carbon disulfide	Merck	EP	>99.5

[a] The purity reported by the supplier. [b] It is also called tetramethylene sulfone, and the CAS registry number of the chemical is [126-33-0].

Apparatus and Procedure. The experimental points that define the binodal curves were determined by using a 50 cm^3 jacketed stainless steel cell controlled at a given temperature. Temperatures were controlled to ± 0.03 K. Mixtures, comprised of (11 to 26) g of sulfolane, (6 to 15) g of heptane, and up to 21 g of toluene or *m*-xylene, were introduced into the cell using pipets and agitated while the temperature was maintained constant with circulation of silicon oil through the external jacket from a thermostatic bath. The sample was stirred for 1 h with a four-blade propeller stirrer at a speed of 1100 rpm and then left to settle for at least 4 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 internal standard, and each sample was diluted with 50 g of carbon disulfide[6] in preparation for analysis. The accuracy of weighing was ± 0.0001 g. The sample analysis was performed using a Hewlett-Packard model 5890 gas chromatograph equipped with a flame ionization detector and a HP Ultra 1 column (cross-linked methyl silicone gum, 25 m $\times$ $3.2 \cdot 10^{-4}$ m $\times$ $5.2 \cdot 10^{-7}$ m film thickness). The injector and detector temperatures were maintained at 563.15 K. The column temperature was programmed for an initial temperature of 348.15 K, maintained for 1 min, and a final temperature of 383.15 K, maintained for 1 min. The heating rate was 5 $K \cdot min^{-1}$, and the flow rate of nitrogen carrier gas was $2.5 \cdot 10^{-5}$ $m^3 \cdot min^{-1}$. Mass fraction measurements were reproducible to within ± 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 2 and 3 for the ternary systems formed by heptane (1) + sulfolane (3) with toluene or *m*-xylene (2), respectively. Concentrations of components i ($i = 1, 2, 3$) in phase L ($L = 1$, raffinate phase;

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Table 2. Experimental LLE Data in Mole Fraction for the System Heptane (1) + Toluene (2) + Sulfolane (3)

	heptane-rich phase			sulfolane-rich phase		
T/K	x_{11}	x_{21}	x_{31}	x_{12}	x_{22}	x_{32}
298.15	0.9992	0.0000	0.0008	0.0048	0.0000	0.9952
	0.8708	0.1244	0.0048	0.0093	0.0383	0.9524
	0.7723	0.2188	0.0089	0.0107	0.0784	0.9109
	0.6954	0.2928	0.0118	0.0120	0.1170	0.8710
	0.5963	0.3842	0.0195	0.0152	0.1724	0.8124
	0.5452	0.4329	0.0219	0.0190	0.2143	0.7667
	0.4453	0.5152	0.0395	0.0226	0.2679	0.7095
	0.2820	0.6141	0.1039	0.0414	0.4183	0.5403
	0.1774	0.6359	0.1867	0.0744	0.5334	0.3922
323.15	0.9958	0.0000	0.0042	0.0120	0.0000	0.9880
	0.8853	0.1091	0.0056	0.0192	0.0336	0.9472
	0.7778	0.2140	0.0082	0.0208	0.0749	0.9043
	0.6644	0.3209	0.0147	0.0229	0.1402	0.8369
	0.5712	0.4032	0.0256	0.0262	0.1898	0.7840
	0.4607	0.4964	0.0429	0.0388	0.2506	0.7106
	0.3693	0.5564	0.0743	0.0545	0.3398	0.6057
	0.3012	0.5893	0.1095	0.0694	0.3986	0.5320
	0.2359	0.5955	0.1686	0.1160	0.4979	0.3861
348.15	0.9925	0.0000	0.0075	0.0203	0.0000	0.9797
	0.9004	0.0830	0.0166	0.0263	0.0250	0.9487
	0.7869	0.1950	0.0181	0.0305	0.0752	0.8943
	0.6936	0.2821	0.0243	0.0324	0.1210	0.8466
	0.5809	0.3786	0.0405	0.0404	0.1808	0.7788
	0.5008	0.4421	0.0571	0.0477	0.2426	0.7097
	0.4043	0.5028	0.0929	0.0614	0.3267	0.6119
	0.3281	0.5429	0.1290	0.0792	0.4011	0.5197
	0.2457	0.5657	0.1886	0.1112	0.4800	0.4088
373.15	0.9898	0.0000	0.0102	0.0322	0.0000	0.9678
	0.8974	0.0921	0.0105	0.0340	0.0301	0.9359
	0.8210	0.1614	0.0176	0.0366	0.0592	0.9042
	0.7271	0.2436	0.0293	0.0410	0.1021	0.8569
	0.6409	0.3129	0.0462	0.0468	0.1487	0.8045
	0.5676	0.3686	0.0638	0.0537	0.1942	0.7521
	0.4977	0.4151	0.0872	0.0637	0.2417	0.6946
	0.4251	0.4568	0.1181	0.0769	0.2964	0.6267
	0.3356	0.4920	0.1724	0.1016	0.3686	0.5298

Table 3. Experimental LLE Data in Mole Fraction for the System Heptane (1) + *m*-Xylene (2) + Sulfolane (3)

	heptane-rich phase			sulfolane-rich phase		
T/K	x_{11}	x_{21}	x_{31}	x_{12}	x_{22}	x_{32}
298.15	0.9992	0.0000	0.0008	0.0048	0.0000	0.9952
	0.8753	0.1229	0.0018	0.0156	0.0299	0.9545
	0.7762	0.2200	0.0038	0.0159	0.0574	0.9267
	0.6798	0.3110	0.0092	0.0160	0.0880	0.8960
	0.5952	0.3910	0.0138	0.0175	0.1173	0.8652
	0.5048	0.4718	0.0234	0.0184	0.1538	0.8278
	0.4155	0.5473	0.0372	0.0196	0.1961	0.7843
	0.3349	0.6074	0.0577	0.0210	0.2402	0.7388
	0.2158	0.6741	0.1101	0.0245	0.3251	0.6504
323.15	0.9958	0.0000	0.0042	0.0120	0.0000	0.9880
	0.8614	0.1334	0.0052	0.0186	0.0334	0.9480
	0.7724	0.2189	0.0087	0.0193	0.0599	0.9208
	0.6872	0.3036	0.0092	0.0202	0.0893	0.8905
	0.5934	0.3953	0.0113	0.0213	0.1249	0.8538
	0.5067	0.4671	0.0262	0.0225	0.1571	0.8204
	0.4321	0.5283	0.0396	0.0239	0.1899	0.7862
	0.3471	0.5876	0.0653	0.0244	0.2339	0.7417
	0.2551	0.6288	0.1161	0.0289	0.2991	0.6720
348.15	0.9925	0.0000	0.0075	0.0203	0.0000	0.9797
	0.9038	0.0829	0.0133	0.0238	0.0208	0.9554
	0.8223	0.1539	0.0238	0.0251	0.0404	0.9345
	0.7440	0.2313	0.0247	0.0269	0.0676	0.9055
	0.6411	0.3336	0.0253	0.0270	0.1079	0.8651
	0.5334	0.4357	0.0309	0.0272	0.1486	0.8242
	0.4452	0.5053	0.0495	0.0283	0.1757	0.7960
	0.3395	0.5840	0.0765	0.0322	0.2411	0.7267
	0.2502	0.6300	0.1198	0.0380	0.3113	0.6507
373.15	0.9898	0.0000	0.0102	0.0322	0.0000	0.9678
	0.8896	0.1001	0.0103	0.0337	0.0343	0.9320
	0.7792	0.2042	0.0166	0.0345	0.0712	0.8943
	0.6832	0.2976	0.0192	0.0362	0.1097	0.8541
	0.5841	0.3879	0.0280	0.0381	0.1452	0.8167
	0.5024	0.4643	0.0333	0.0390	0.1972	0.7638
	0.4214	0.5286	0.0500	0.0435	0.2279	0.7286
	0.3393	0.5821	0.0786	0.0512	0.2723	0.6765
	0.2662	0.6210	0.1128	0.0516	0.3334	0.6150

$L = 2$, extract phase) are given in mole fraction, x_{iL}. The effect of temperature on the equilibrium for the system heptane (1) + toluene (2) + sulfolane (3) is shown in Figure 1. As seen, the size of the two-phase region decreases with an increase in temperature. A similar trend was observed for the other system containing *m*-xylene. These systems studied present a wide two-phase region which is important, together with the slope of the tie lines, when establishing the selectivity of sulfolane.

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 the tie lines for each ternary system using the Simplex Search method. The objective function (F) used is

$$F = \min \sum_{i} \sum_{L} \sum_{k} (x_{iLk} - x^{c})^{2} \qquad (1)$$

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.

For the NRTL model, the third nonrandomness parameter, α_{ij}, was set at a value of 0.2.[8] The parameters calculated are shown in Tables 4 and 5, together with the root-mean-square deviation (rmsd) values, defined as

$$\mathrm{rmsd} = 100\left[\sum \sum \sum (x_{iLk} - x^{c})^{2}/6N\right]^{1/2} \qquad (2)$$

where N = number of tie lines.

The rmsd is a measure of the agreement between the experimental data and the calculated values. As can be seen from Tables 4 and 5, the calculation based on both the UNIQUAC and the NRTL models gave a 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 the rmsd (the average root-mean-square deviation between the phase composi-

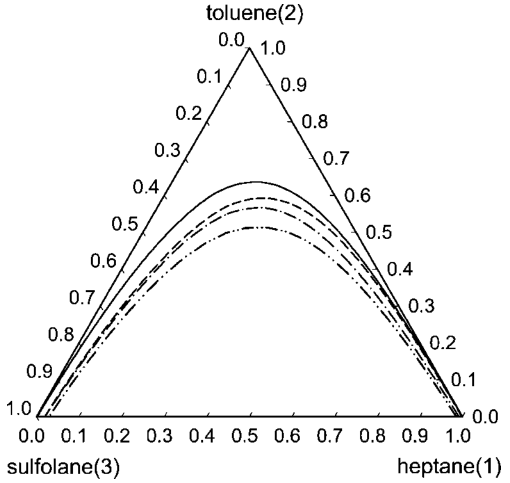

Figure 1. Effect of temperature on the liquid–liquid equilibrium for the heptane (1) + toluene (2) + sulfolane (3) system. Curves calculated by UNIQUAC: —, 298.15 K; ---, 323.15 K; —•—, 348.15 K; −••−, 373.15 K.

Table 4. UNIQUAC and NRTL Parameters for the System Heptane (1) + Toluene (2) + Sulfolane (3) at (298.15, 323.15, 348.15, and 373.15) K, as Well as the Calculated Root-Mean-Square Deviation (rmsd)

		UNIQUAC parameters/K			NRTL parameters/K		
T/K	*i*–*j*	$(u_{ij} - u_{jj})/R$	$(u_{ji} - u_{ii})/R$	rmsd	$(g_{ij} - g_{jj})/R$	$(g_{ji} - g_{ii})/R$	rmsd
298.15	1–2	404.49	−272.30	0.4551	−625.52	675.12	0.6107
	1–3	559.49	181.52		1304.00	823.97	
	2–3	214.96	−87.91		262.48	−12.00	
323.15	1–2	366.69	−314.36	0.9493	87.66	−363.20	1.0984
	1–3	657.57	14.87		1191.10	976.98	
	2–3	228.41	−96.71		671.13	−253.65	
348.15	1–2	268.11	−255.34	0.3835	−570.48	−24.36	0.7096
	1–3	473.55	61.60		1181.10	927.24	
	2–3	300.92	−136.74		368.28	−339.50	
373.15	1–2	251.62	−272.17	0.5848	−520.85	−196.82	0.6442
	1–3	464.31	48.56		1302.20	753.22	
	2–3	283.74	−142.96		305.60	−326.28	

Table 5. UNIQUAC and NRTL Parameters for the System Heptane (1) + *m*-Xylene (2) + Sulfolane (3) at (298.15, 323.15, 348.15, and 373.15) K, as Well as the Calculated Root-Mean-Square Deviation (rmsd)

		UNIQUAC parameters/K			NRTL parameters/K		
T/K	*i*–*j*	$(u_{ij} - u_{jj})/R$	$(u_{ji} - u_{ii})/R$	rmsd	$(g_{ij} - g_{jj})/R$	$(g_{ji} - g_{ii})/R$	rmsd
298.15	1–2	−240.49	429.08	0.2837	561.12	−444.87	0.3922
	1–3	612.38	44.72		1467.00	888.46	
	2–3	206.84	−13.67		687.72	−8.86	
323.15	1–2	−276.49	562.25	0.4671	282.99	−269.65	0.6406
	1–3	582.74	25.58		1274.90	824.73	
	2–3	268.77	−29.09		687.39	6.76	
348.15	1–2	91.06	−100.65	0.4164	162.32	−233.69	0.5216
	1–3	547.26	26.83		1150.30	842.82	
	2–3	214.80	−19.92		708.43	0.70	
373.15	1–2	−28.56	42.43	0.5488	138.26	−105.02	0.5630
	1–3	578.91	0.94		1321.80	737.51	
	2–3	255.23	−36.73		744.08	−15.82	

tion obtained from experiment and that from calculation was 0.5111 for UNIQUAC as compared to 0.6475 for NRTL).

Discussion

The calculated values using both the UNIQUAC and NRTL models for the heptane (1) + *m*-xylene (2) + sulfolane (3) system at (298.15, 323.15, 348.15, and 373.15) K are compared with the experimental data in Figures 2 to 5. It was observed that the slopes of the tie lines show that *m*-xylene is more soluble in heptane than in sulfolane. It is observed that the relative mutual solubility of toluene is higher than that of *m*-xylene in heptane + sulfolane mixtures at the same temperature. This solubility effect is reflected in the size of the two-phase region, increasing slightly in the order *m*-xylene > toluene at the same temperature. All the ternary systems heptane (1) + (toluene or *m*-xylene) (2) + sulfolane (3) at any given temperature of this study are type 1 systems.[19]

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 heptane

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

where the subscript 2 represents toluene or *m*-xylene and 1 represents heptane.

This quantity is not constant over the whole two-phase region. Table 6 lists experimental values of *S*. From the data, we find

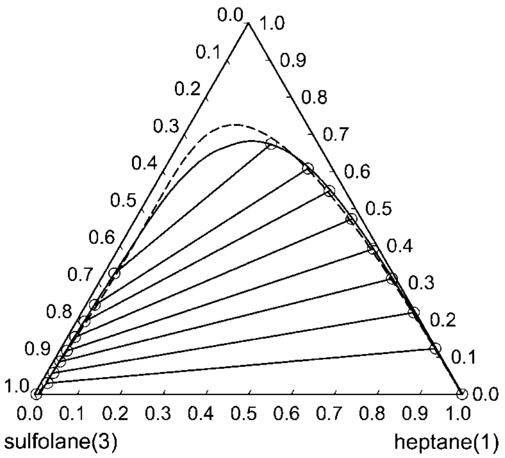

Figure 2. LLE data in mole fraction for the heptane (1) + *m*-xylene (2) + sulfolane (3) system at 298.15 K. Curves calculated by —, UNIQUAC model; ---, NRTL model; ○-○, experimental tie line.

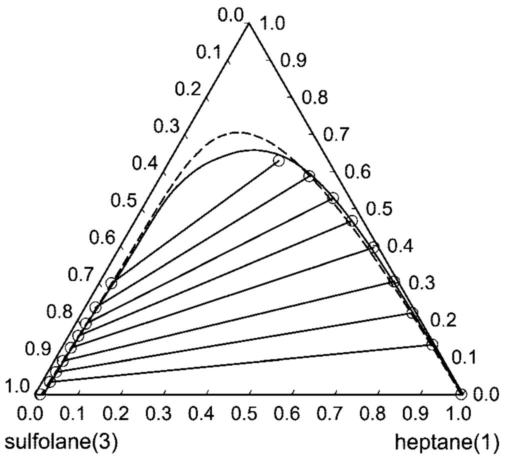

Figure 3. LLE data in mole fraction for the heptane (1) + *m*-xylene (2) + sulfolane (3) system at 323.15 K. Curves calculated by —, UNIQUAC model; ---, NRTL model; ○-○, experimental tie line.

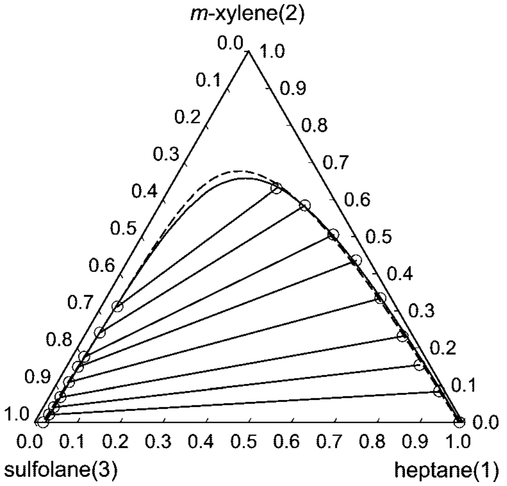

Figure 4. LLE data in mole fraction for the heptane (1) + *m*-xylene (2) + sulfolane (3) system at 348.15 K. Curves calculated by —, UNIQUAC model; ---, NRTL model; ○-○, experimental tie line.

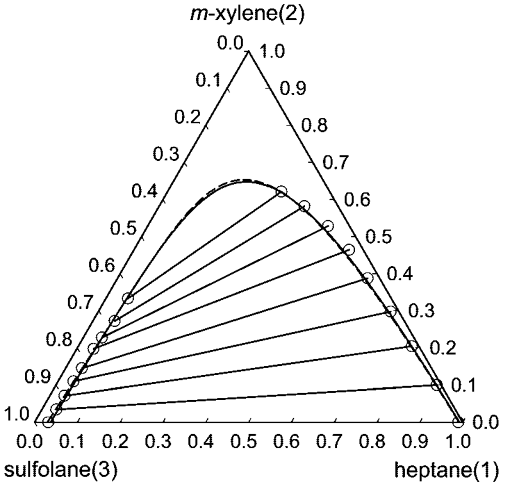

Figure 5. LLE data in mole fraction for the heptane (1) + *m*-xylene (2) + sulfolane (3) system at 373.15 K. Curves calculated by —, UNIQUAC model; ---, NRTL model; ○-○, experimental tie line.

Table 6. Experimental Selectivity Values *S* for Each Tie Line

S for the system	298.15 K	323.15 K	348.15 K	373.15 K
toluene	28.8	14.2	10.3	8.6
	25.9	13.1	9.9	8.2
	23.2	12.7	9.2	7.4
	17.6	10.3	6.9	6.5
	14.2	6.0	5.8	5.6
	10.2	4.1	4.3	4.5
	4.6	2.9	3.1	3.6
	2.0	1.7	1.9	2.5
m-xylene	13.7	11.6	9.5	9.0
	12.7	11.0	8.6	7.9
	12.0	10.0	8.1	7.0
	10.2	8.8	7.7	5.7
	8.9	7.6	6.7	5.5
	7.6	6.5	5.5	4.2
	6.3	5.7	4.4	3.1
	4.2	4.2	3.3	2.8

Table 7. Experimental Distribution Coefficient Values κ for Each Tie Line

κ for the system	298.15 K	323.15 K	348.15 K	373.15 K
toluene	0.308	0.308	0.301	0.327
	0.358	0.350	0.386	0.367
	0.400	0.437	0.429	0.419
	0.449	0.471	0.478	0.475
	0.495	0.505	0.549	0.527
	0.520	0.611	0.650	0.582
	0.681	0.676	0.739	0.649
	0.839	0.836	0.849	0.750
m-xylene	0.243	0.250	0.251	0.343
	0.261	0.274	0.263	0.349
	0.283	0.294	0.292	0.369
	0.300	0.316	0.323	0.374
	0.326	0.336	0.341	0.425
	0.358	0.360	0.348	0.431
	0.396	0.398	0.413	0.468
	0.482	0.476	0.494	0.537

the tendency that, for the same system, the higher the temperature, the lower the selectivity. In the temperature range (298.15 to 348.15) K, for the different systems at the same temperature, the order of the selectivity of sulfolane to aromatic is toluene > *m*-xylene. Hence, sulfolane is more selective to lower carbon number than to higher carbon number in aromatic hydrocarbons. As seen, *S* decreases when going through the tie lines from low concentration to high concentration of aromatic. It means the higher the concentration of aromatic in the feed, the lower the selectivity of sulfolane to aromatic. Since the selectivity in all cases is greater than 1, it means that extraction is possible.

The capacity or dissolving ability of a solvent is a measure of the ratio of solvent to feed required for the desired recovery of aromatics. The distribution coefficient for aromatic, which is the measure of the solvent power or capacity of sulfolane, is given as

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

The experimental values of κ of this study are presented in Table 7. We can find that, for the same system, there are not obvious differences between the κ values in the temperature range of (298.15 to 373.15) K. At the same temperature for the different systems, the κ values for the system increase in the order of toluene > *m*-xylene. This result is similar to the trend of *S* values.

In this work, sulfolane shows a greater selectivity and capacity for the aromatic hydrocarbons in the order of toluene > *m*-xylene, indicating that the extractive ability of sulfolane decreases with an increasing number of alkyl (methyl) groups on the aromatic compound. This is because the difference of the steric interference effect exists in a different number of methyl groups on aromatic hydrocarbons.

Conclusions

Liquid–liquid equilibrium data of the ternary systems heptane (1) + toluene (2) + sulfolane (3) and heptane (1) + *m*-xylene (2) + sulfolane (3) were determined at (298.15, 323.15, 348.15, and 373.15) K, respectively.

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 or NRTL model for the systems studied here show that the size of the two-phase region decreases with increasing temperature. For the same system, the higher the temperature, the lower the selectivity. From the selectivity values, the separation of toluene or *m*-xylene from heptane by extraction with sulfolane is feasible. Not only the selectivity data but also the distribution coefficient data showed that sulfolane is more extractive for toluene than for *m*-xylene at a given temperature.

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