Cosolvent Selection for Benzene−Cyclohexane Separation in Extractive Distillation

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Cosolvent Selection for Benzene−Cyclohexane Separation in Extractive Distillation

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The Krupp−Koppers (K-K) extractive distillation method with *N*-formylmorpholine (NFM) as the solvent is one of the most important processes for catalytic hydrogen refining of rude benzene. To increase the capacity and selectivity of the solvent and decrease the ratio of solvent to feed in this process, ethylene glycol (EG), *N,N*-dimethylformamide (DMF), and *N*-methylpyrrolidone (NMP) were introduced as the cosolvent with NFM. The relative volatility of cyclohexane to benzene (α) at a certain R_{stf} (liquid-phase ratio of solvent to feed) was considered as a criterion of the performance of cosolvent. The vapor−liquid equilibrium (VLE) data for benzene + NFM, EG + NFM, benzene + cyclohexane + NFM, benzene + cyclohexane + NFM + EG, benzene + cyclohexane + NFM + DMF, and benzene + cyclohexane + NFM + NMP were measured at atmospheric pressure. VLE data of benzene + NFM, EG + NFM, benzene + cyclohexane + NFM, and benzene + cyclohexane + NFM + EG were calculated by the nonrandom two-liquid (NRTL) model. The average temperature deviations of benzene + cyclohexane + NFM and benzene + cyclohexane + NFM + EG systems are (1.40 and 3.19) K, respectively. The average deviations of the vapor-phase mole fraction of benzene are 0.030 and 0.066, respectively.

Introduction

Catalytic hydrogen refining of rude benzene is an important method for the production of pure benzene. Extractive distillation is one of the most important unit processes in refining. In the extractive distillation methods, the Krupp−Koppers (K-K) extractive distillation method is widely used with *N*-formylmorpholine (NFM) as the solvent. In some projects, it is a bottleneck to increase the throughput of pure benzene by decreasing the ratio of solvent to feed.

The utilization of cosolvent for extractive distillation drew considerable attention in the past decade. Lee and Brown[1−4] have studied the composition of cosolvent for the separation of a cycloalkane and close-boiling alkane and proposed several compositions of cosolvents, while NFM was not included in their study. In this paper, ethylene glycol (EG), *N,N*-dimethylformamide (DMF), and *N*-methylpyrrolidone (NMP) were selected as the second solvent to form a cosolvent with NFM. Considering the composition of the refined rude benzene and the close boiling points of benzene and cyclohexane, the feed was determined as benzene and cyclohexane with a mass fraction 10:1. The vapor−liquid equilibrium (VLE) data for two binary systems of benzene + NFM and EG + NFM, the ternary system of benzene + cyclohexane + NFM, and three quaternary systems of benzene + cyclohexane + NFM + EG, benzene + cyclohexane + NFM + DMF, and benzene + cyclohexane + NFM + NMP were measured at 101.32 kPa. Equations 1 and 2 were used to calculate α_{ij} (the relative volatility of cyclohexane to benzene) and R_{stf}, respectively.

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$$\alpha_{ij} = \frac{y_i/x_i}{y_j/x_j} \tag{1}$$

where the subscripts *i* and *j* refer to cyclohexane and benzene in this study; *x* and *y* are the mole fractions of liquid-phase and vapor-phase, respectively.

$$R_{stf} = w_s/w_f \tag{2}$$

In eq 2, w_s is the liquid-phase mass of solvent, and w_f is the mass of the feed.

Experimental Section

Materials. NFM was supplied by Shijiazhuang Coking Group Co., Ltd. All of the other chemicals were supplied by Tianjin Reagent Co. NFM was purified by distillation, while the other chemicals were subjected to no further purification. The purities tested by gas chromatography were as follows: NFM (> 0.995), benzene (> 0.995), cyclohexane (> 0.995), EG (> 0.997), DMF (> 0.995), and NMP (> 0.995).

Apparatus and Procedure. The mass fractions of the cosolvents selected including EG, DMF, and NMP were as follows: EG, 0.1000 and 0.2000; DMF, 0.1000 and 0.2000; NMP, 0.2000. The mixtures were gravimetrically measured by an electronic balance (Sartorius BS224, Germany) accurate to within ± 0.0001 g.

The VLE data were measured in a modified Rose equilibrium still, which was shown in Figure 1. In this still, both the vapor and the liquid phases were continuously recirculating to provide intimate contact of the phases and ensure that equilibrium could be established rapidly. The chemicals were added into the still at the beginning of the experiment. Equilibrium was assumed when constant temperature had been kept for more than 30 min, and then samples were taken by microliter syringes (0.2 μL for
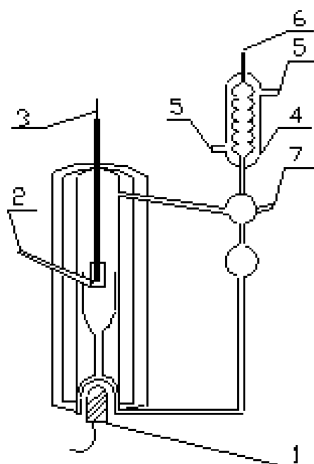
Figure 1. Improved Rose equilibria still: 1, heating column; 2, liquid sample connection; 3, a precise mercury thermometer; 4, condenser; 5, condensate; 6, contact with pressure-controlling devices; 7, vapor (cooled to liquid) sample connection.

Table 1. VLE Data for the Binary Systems at 101.32 kPa

no.	T/K	x_1	x_2	y_1	y_2
		Benzene (1) + NFM (2)			
1	355.00	0.9207	0.0793	0.9994	0.0006
2	355.75	0.8792	0.1208	0.9991	0.0009
3	357.46	0.8296	0.1704	0.9989	0.0011
4	358.74	0.7487	0.2513	0.9986	0.0014
5	360.40	0.7078	0.2922	0.9983	0.0017
6	363.45	0.6154	0.3846	0.9968	0.0032
7	366.85	0.5514	0.4486	0.9965	0.0035
8	370.85	0.4717	0.5283	0.9910	0.0090
9	377.85	0.3887	0.6113	0.9879	0.0121
10	388.35	0.2900	0.7100	0.9854	0.0146
		EG (1) + NFM (2)			
1	469.15	1.0000	0.0000	1.0000	0.0000
2	470.54	0.9560	0.0440	0.9827	0.0173
3	470.77	0.9403	0.0597	0.9790	0.0210
4	471.35	0.8935	0.1065	0.9621	0.0379
5	471.55	0.8896	0.1104	0.9619	0.0381
6	472.15	0.8690	0.1310	0.9559	0.0441
7	473.65	0.8335	0.1665	0.9410	0.0590
8	474.48	0.7966	0.2034	0.9296	0.0704
9	476.40	0.7315	0.2685	0.9022	0.0978
10	477.75	0.6856	0.3144	0.8825	0.1175
11	478.80	0.6354	0.3646	0.8759	0.1241
12	480.85	0.5693	0.4307	0.8320	0.1680
13	484.60	0.4791	0.5209	0.7563	0.2437
14	488.80	0.3720	0.6280	0.6796	0.3204
15	489.45	0.3527	0.6473	0.6697	0.3303
16	497.55	0.1818	0.8182	0.4351	0.5649
17	501.25	0.1202	0.8798	0.3113	0.6887
18	509.95	0.0000	1.0000	0.0000	1.0000

Table 2. VLE Data for the Benzene (1) + Cyclohexane (2) + NFM (3) System at 101.32 kPa

no.	T/K	x_1	x_2	x_3	y_1	y_2	y_3	R_{stf}	α
1	356.95	0.7284	0.0719	0.1997	0.8458	0.1540	0.0002	0.37	1.84
2	358.35	0.6748	0.0723	0.2529	0.8388	0.1612	0.0000	0.50	1.79
3	361.97	0.5657	0.0481	0.3862	0.8293	0.1700	0.0007	0.92	2.41
4	362.55	0.5502	0.0492	0.4006	0.8247	0.1738	0.0015	0.98	2.36
5	368.25	0.4223	0.0306	0.5471	0.8268	0.1716	0.0016	1.77	2.86
6	376.05	0.3475	0.0210	0.6315	0.8319	0.1657	0.0024	2.51	3.29
7	376.67	0.3538	0.0227	0.6235	0.8253	0.1718	0.0029	2.43	3.24
8	383.13	0.2670	0.0133	0.7197	0.8457	0.1503	0.0040	3.77	3.57
9	392.41	0.2156	0.0098	0.7746	0.8489	0.1458	0.0053	5.05	3.79
10	402.85	0.1553	0.0068	0.8379	0.8461	0.1433	0.0106	7.59	3.88

each time). The equilibrium temperature was measured by a calibrated thermometer with an uncertainty of $\pm$ 0.01 K. The pressure of the system was determined by a U-shaped differential manometer whose fluctuation was held within 0.03 kPa with a two-step automatic control system.

The vapor (cooled to liquid) and liquid samples were analyzed by a gas chromatograph (GC112A supplied by Shanghai Precision & Scientific Instrument Co. Ltd. Factory) with a capillary column (OV-1701 supplied by Ke Kaidi in Lanzhou Chemical Industry New Technology Co. Ltd.) and a flame ionization detector (FID). High-purity nitrogen (0.9999) was taken as the carrier gas. Each sample was analyzed at least thrice to ensure the accuracy, and the uncertainty of the mole fractions were $\pm$ 0.0001.

The reliability of the experimental system has been tested in our previous work.[5] The result shows a good reliability of the system.

Results and Discussion

Results of Measurement. The VLE data for benzene + NFM and EG + NFM binary systems, the benzene + cyclohexane + NFM ternary system, and benzene + cyclohexane + NFM + EG (mass fraction of EG is 0.1000), benzene + cyclohexane + NFM + EG (mass fraction of EG is 0.2000), benzene + cyclohexane + NFM + DMF (mass fraction of DMF is 0.1000), benzene + cyclohexane + NFM + DMF (mass fraction of DMF is 0.2000), and benzene + cyclohexane + NFM + NMP (mass fraction of NMP is 0.2000) quaternary systems are given in Tables 1 to 3. The corresponding α and R_{stf} are also presented in these tables.

The $T-x-y$ plots of benzene + NFM and EG + NFM systems are shown in Figures 2 and 3. The plots of α against R_{stf} in different cosolvent systems as well as that in NFM as the only solvent system are given in Figure 4.

Figure 4 shows that, at low R_{stf}, α increases rapidly as R_{stf} is increasing. When R_{stf} reaches a certain value and continues to increase, α appears to have no obvious change. These values of R_{stf} are considered to be the suitable ratio of solvent to feed for the corresponding cosolvent. For example, in the benzene + cyclohexane + NFM system, when R_{stf} reaches 5.05, α reaches 3.79 and appears to have no obvious changes while R_{stf} is increasing, so the suitable R_{stf} for NFM is 5.05. It can also be observed that α increased when EG (mass fraction of

EG is 0.2000) was added to the ternary system of benzene + cyclohexane + NFM. The value of R_{stf} is 3.67, while α reaches 3.73. That is to say, when α reaches 3.73, which has no obvious improvement in both benzene + cyclohexane + NFM and benzene + cyclohexane + NFM + EG (mass fraction of EG is 0.2000) systems, R_{stf} decreases to almost 1.38 when EG (mass fraction of EG is 0.2000) is added, while in the other quaternary systems, R_{stf} fails to decrease compared to the benzene + cyclohexane + NFM ternary system.

Results of Correlation. The NRTL model (shown in Table 4) was used to correlate the binary VLE data of benzene + NFM and EG + NFM and the binary liquid−liquid equilibrium (LLE) data of benzene + EG (LLE data are from the literature[6−8]). The objective functions for correlating are F_1 and F_2, respectively:

$$F_1 = \frac{1}{n}\sum_{i=1}^{n}\left[\left|\frac{y_{1,cal} - y_{1,exp}}{y_{1,exp}}\right|_i + \left|\frac{T_{cal} - T_{exp}}{T_{exp}}\right|_i\right] \quad (3)$$

$$F_2 = \frac{1}{n}\sum_{i=1}^{n}\left\{\left|\frac{x_{1,cal} - x_{1,exp}}{x_{1,exp}}\right|_i + \left|\frac{x_{2,cal} - x_{2,exp}}{x_{2,exp}}\right|_i\right\} \quad (4)$$

where y_1 is the vapor-phase mole fraction of component 1; T is the equilibrium temperature (K); x_1 and x_2 are the mole fractions

Table 3. VLE Data for the Quaternary Systems at 101.32 kPa

no.	T/K	x_1	x_2	x_3	y_1	y_2	y_3	R_{stf}	α
				Benzene (1) + Cyclohexane (2) + NFM (3) + EG (4)					
				Mass Fraction of EG = 0.1000					
1	355.76	0.7481	0.0766	0.1482	0.8463	0.1503	0.0002	0.29	1.74
2	358.63	0.6193	0.0564	0.2731	0.8325	0.1633	0.0004	0.65	2.15
3	362.33	0.5006	0.0359	0.3883	0.8335	0.1620	0.0007	1.17	2.71
4	367.85	0.3856	0.0241	0.4939	0.8266	0.1635	0.0011	1.96	3.17
5	375.95	0.2823	0.0173	0.6008	0.8093	0.1722	0.0027	3.59	3.69
6	379.35	0.2657	0.0141	0.6237	0.8323	0.1588	0.0026	3.91	3.71
7	382.95	0.2478	0.0127	0.6394	0.8299	0.1578	0.0028	5.09	3.80
8	383.35	0.2411	0.0124	0.6460	0.8205	0.1549	0.0021	4.39	3.74
9	386.15	0.2026	0.0101	0.6786	0.8097	0.1535	0.0040	4.94	3.84
10	386.35	0.2280	0.0109	0.6577	0.8363	0.1496	0.0030	5.49	3.78
				Mass Fraction of EG = 0.2000					
1	354.83	0.7458	0.0767	0.1255	0.8345	0.1448	0.0002	0.27	1.69
2	355.45	0.7220	0.0714	0.1528	0.8510	0.1436	0.0000	0.34	1.71
3	359.90	0.4819	0.0394	0.3316	0.8154	0.1695	0.0004	1.16	2.54
4	365.70	0.3484	0.0200	0.4625	0.8388	0.1536	0.0009	2.21	3.19
5	373.70	0.2653	0.0125	0.5303	0.8455	0.1417	0.0022	3.35	3.57
6	374.75	0.2475	0.0125	0.5445	0.8331	0.1577	0.0025	3.67	3.73
7	381.55	0.2081	0.0091	0.5789	0.8444	0.1435	0.0042	4.66	3.91
8	386.15	0.1891	0.0074	0.5899	0.8501	0.1321	0.0047	5.27	3.95
9	388.17	0.1683	0.0073	0.6012	0.8298	0.1438	0.0055	6.04	4.00
10	392.40	0.1503	0.0059	0.6234	0.8399	0.1327	0.0100	6.99	4.04
11	395.45	0.1405	0.0049	0.6245	0.8432	0.1193	0.0122	7.57	4.05
				Benzene (1) + Cyclohexane (2) + NFM (3) + DMF (4)					
				Mass Fraction of DMF = 0.1000					
1	357.35	0.7070	0.0728	0.1865	0.8403	0.1553	0.0000	0.39	1.80
2	361.72	0.5574	0.0511	0.3334	0.8196	0.1720	0.0008	0.89	2.29
3	370.12	0.3989	0.0284	0.4850	0.8146	0.1686	0.0019	1.86	2.91
4	386.81	0.2412	0.0124	0.6389	0.8134	0.1417	0.0061	4.10	3.40
5	400.03	0.1724	0.0085	0.6977	0.7968	0.1422	0.0073	6.29	3.61
				Mass Fraction of DMF = 0.2000					
1	359.07	0.6572	0.0655	0.1969	0.8267	0.1624	0.0000	0.50	1.97
2	368.92	0.4278	0.0323	0.3845	0.8039	0.1661	0.0015	1.54	2.74
3	383.72	0.2598	0.0153	0.5204	0.7848	0.152	0.0035	3.47	3.29
4	398.15	0.1825	0.0085	0.5679	0.7579	0.1227	0.0084	5.54	3.47
				Benzene (1) + Cyclohexane (2) + NFM (3) + NMP (4)					
				Mass Fraction of NMP = 0.2000					
1	361.90	0.0485	0.5891	0.1764	0.1620	0.8359	0.0018	0.94	2.36
2	373.00	0.0266	0.4038	0.2824	0.1649	0.8314	0.0038	2.18	3.01
3	379.67	0.0198	0.3550	0.3111	0.1553	0.8395	0.0057	2.76	3.32
4	385.40	0.0156	0.2969	0.3414	0.1497	0.8387	0.0124	3.64	3.39
5	390.00	0.0120	0.2509	0.3655	0.1457	0.8405	0.0153	4.64	3.64
6	393.87	0.0106	0.2199	0.3805	0.1507	0.8327	0.0176	5.52	3.76
7	409.20	0.0063	0.1522	0.4127	0.1308	0.8236	0.0482	8.77	3.82

of component 1 and 2 in liquid-phase, respectively; n is the number of experimental points; and the subscripts cal and exp represent the calculated and experimental values, respectively.
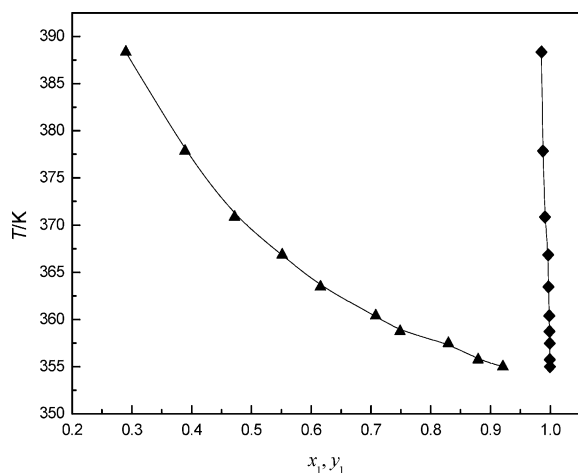

Figure 2. $T-x-y$ for the benzene (1) + NFM (2) system at atmospheric pressure. T, temperature; x_1, liquid-phase mole fraction; y_1, vapor-phase mole fraction. Temperature vs vapor-phase mole fraction is shown as ◆. Temperature vs liquid-phase mole fraction is shown as ▲.
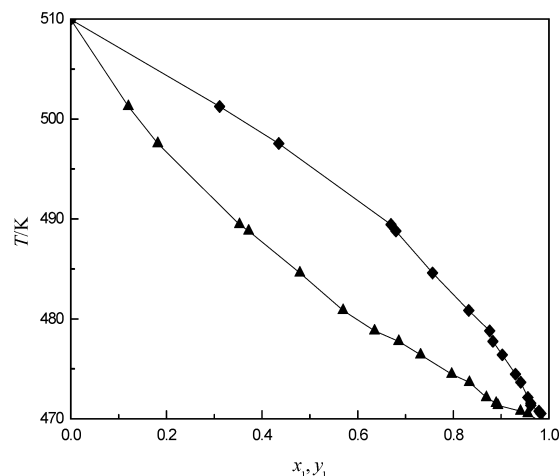

Figure 3. Temperature vs liquid and vapor-phase mole fraction for the EG (1) + NFM (2) system at atmospheric pressure. T, temperature; x_1, liquid-phase mole fraction; y_1, vapor-phase mole fraction. Temperature vs vapor-phase mole fraction is shown as ◆. Temperature vs liquid-phase mole fraction is shown as ▲.
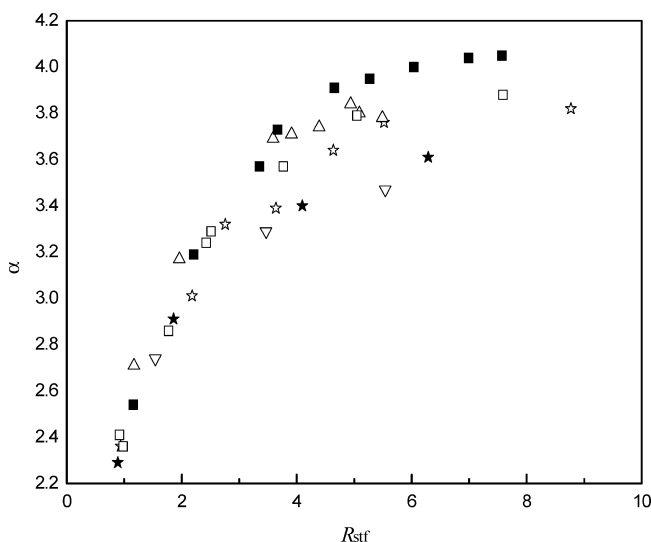

Figure 4. Relative volatility of cyclohexane to benzene vs the ratio of solvent to feed for systems. ☆, benzene (1) + cyclohexane (2) + NFM (3) + NMP (4) (mass fraction of NMP is 0.2000); △, benzene (1) + cyclohexane (2) + NFM (3) + EG (4) (mass fraction of EG is 0.1000); ■, benzene (1) + cyclohexane (2) + NFM (3) + EG (4) (mass fraction of EG is 0.2000); □, benzene (1) + cyclohexane (2) + NFM (3); ★, benzene (1) + cyclohexane (2) + NFM (3) + DMF (4) (mass fraction of DMF is 0.1000); ▽, benzene (1) + cyclohexane (2) + NFM (3) + DMF (4) (mass fraction of DMF is 0.2000).

The NRTL model was used to predict the VLE equilibrium data for the ternary system of benzene + cyclohexane + NFM and quaternary systems of benzene + cyclohexane + NFM + EG (mass fraction of EG is 0.2000). All of the interaction parameters for the NRTL model are listed in Table 4. The interaction parameters for benzene + cyclohexane, EG + benzene, cyclohexane + EG, and cyclohexane + NFM binary systems were obtained from literature.[6−8] The parameters for EG + NFM and benzene + NFM binary systems were calculated from Table 1. The Antoine constants for the components used in the calculations were cited from literature.[9,10] The deviations between the calculated and the experimental results of the vapor-phase mole fraction Δy_1 and temperature ΔT of the benzene + NFM, EG + NFM, benzene + cyclohexane + NFM, and benzene + cyclohexane + NFM + EG (mass fraction of EG is 0.2000) systems are listed in Table 5.

Table 4. Parameters of the NRTL Model^a for Binary Systems

system	A_{12}	A_{21}	α	
benzene (1) + cyclohexane (2)[6]	1528.91 J·mol^{-1}	-445.42 J·mol^{-1}	0.30	$A_{ij} = (g_{ij} - g_{jj})$
benzene (1) + NFM (2)	6157.23 J·mol^{-1}	-231.92 J·mol^{-1}	0.33	$A_{ij} = (g_{ij} - g_{jj})$
EG (1) + benzene (2)[7]	5396.76 J·mol^{-1}	3691.95 J·mol^{-1}	0.18	$A_{ij} = (g_{ij} - g_{jj})$
cyclohexane (1) + EG (2)[7]	2417.1 K	1387 K	0.2	$A_{ij} = (g_{ij} - g_{jj})/R$
EG (1) + NFM (2)	446.19 J·mol^{-1}	264.71 J·mol^{-1}	0.29	$A_{ij} = (g_{ij} - g_{jj})$
cyclohexane (1) + NFM (2)[8]	$\tau_{ij} = a_{ij} + b_{ij}/T + e_{ij} \ln T$		$\alpha_{ij} = c_{ij} + d_{ij}(T - 273.15)$	
	where $a_{12} = 20.719$		$a_{21} = 120.47$	
	$b_{12}/K = 900.64$		$b_{21}/K = -4921.0$	
	$c_{12} = c_{21} = 0.3$			
	$d_{12}/K^{-1} = 0.0001$		$d_{21}/K^{-1} = 0$	
	$e_{12} = -3.536$		$e_{21} = -17.927$	

^{a} NRTL model:

$$\ln \gamma_1 = x_2^2 \left[\frac{\tau_{21}G_{21}^2}{(x_1 + x_2 G_{21})^2} + \frac{\tau_{12}G_{12}}{(x_2 + x_1 G_{12})^2} \right]$$

$$\ln \gamma_2 = x_1^2 \left[\frac{\tau_{12}G_{12}^2}{(x_2 + x_1 G_{12})^2} + \frac{\tau_{21}G_{21}}{(x_1 + x_2 G_{21})^2} \right]$$

$$\tau_{12} = \frac{g_{12} - g_{22}}{RT} \qquad \tau_{21} = \frac{g_{21} - g_{11}}{RT}$$

$$G_{12} = \exp(-\alpha_{12}\tau_{12}) \qquad G_{21} = \exp(-\alpha_{21}\tau_{21})$$

Table 5. Deviations between the Calculated and the Experimental Results of the Five Systems Using the NRTL Equation^a

system		Δy_1	$\Delta T/K$
benzene (1) + NFM (2)	ave^b	0.010	0.34
	max^c	0.019	0.72
EG (1) + NFM (2)	ave	0.022	0.90
	max	0.043	1.78
benzene (1) + cyclohexane (2) + NFM (3)	ave	0.030	1.40
	max	0.081	4.47
benzene (1) + cyclohexane (2) + NFM (3) + EG (4)	ave	0.066	3.19
	max	0.010	4.69
		Δx_1	Δx_2
benzene (1) + EG (2)	ave	0.026	0.012
	max	0.060	0.037

^{a}

$$\Delta x_{i,\text{ave}} = \frac{1}{n}\sum_{j=1}^{n} |x_{ij,\text{cal}} - x_{ij,\text{exp}}| \qquad \Delta x_{i,\text{max}} = \max\{|x_{ij,\text{cal}} - x_{ij,\text{exp}}|\}$$

$$i = 1, 2 \quad j = 1, 2,...,n$$

$$\Delta y_{1,\text{ave}} = \frac{1}{n}\sum_{j=1}^{n} |y_{1j,\text{cal}} - y_{1j,\text{exp}}| \qquad \Delta y_{1,\text{max}} = \max\{|y_{1j,\text{cal}} - y_{1j,\text{exp}}|\}$$

$$j = 1, 2,...,n$$

$$\Delta T_{\text{ave}} = \frac{1}{n}\sum_{j=1}^{n} |T_{j,\text{cal}} - T_{j,\text{exp}}| \qquad \Delta T_{\text{max}} = \max\{|T_{j,\text{cal}} - T_{j,\text{exp}}|\}$$

$$j = 1, 2,...,n$$

^{b} ave, the average value. ^{c} max, the maximum value.

The mole fraction deviations between the calculated and the experimental results of component 1 Δx_1 and component 2 Δx_2 of the benzene + EG system are also given in Table 5. The results indicate that the correlated results are satisfactory and the predictions are acceptable for engineering design.

Conclusion

VLE data for multicomponent mixtures containing NFM at a pressure of 101.32 kPa were measured in this study. The NRTL model was used to predict the VLE data for the benzene + cyclohexane + NFM and benzene + cyclohexane + NFM + EG (mass fraction of EG is 0.2000) systems. The average deviations of temperature are (1.40 and 3.19) K, respectively. The average deviations of the vapor-phase mole fraction of benzene are 0.030 and 0.066, respectively.

The experimental data show that, when EG with a mass fraction of 0.1000, DMF with mass fraction of 0.1000 and 0.2000, and NMP with a mass fraction of 0.2000 were added into NFM, the ratio of solvent to feed would be higher. The cosolvent composed by EG and NFM with a mass proportion of 2:8 (mass fraction of EG is 0.2000) can obtain a lower ratio of solvent to feed than NFM as the only solvent.

According to the results, the ratio of solvent to feed can be decreased by the addition of EG with a suitable mass fraction in the K-K extractive distillation process.

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