Boiling points for five binary systems of sulfolane with aromatic hydrocarbons at 101.33 kPa

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Abstract

Boiling points have been determined at 101.33 kPa for the binary mixtures of sulfolane + o-xylene, sulfolane + m-xylene, sulfolane+p-xylene, sulfolane+ethylbenzene and sulfolane+1,2,4-trimethylbenzene. Calculations of the non-ideality of the vapor phase were made with the second virial coefficients evaluated from the Hayden–O'Connell method. The binary parameters for five activity coefficient models (Margules, van Laar, Wilson, NRTL and UNI-QUAC) have been fitted with the experimental boiling points measured in this work. A comparison of model performances has been carried out using the criterion of the average absolute deviations in boiling point. The activity coefficient of the component in the liquid phase is discussed based on the UNIFAC model with the consideration of the dipole–dipole interactions. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Experiments; Vapor–liquid equilibrium; Binary system; Sulfolane; Aromatic hydrocarbon

1. Introduction

Solvent extraction is one of the most important methods used to produce high-purity aromatic extracts from catalytic reformates, and sulfolane is one of the most widely used solvents in this process. Although sulfolane has been used successfully in large-scale separation plants (SHELL process), not many experimental data are published for typical mixtures, especially not complete sets of data for all binary vapor–liquid equilibria. The basic physical data for the systems involved in this extraction process are required to optimize the design.

Jannelli and Sacco [1] investigated the viscosity, dielectric constant and solid–liquid equilibria for the sulfolane+benzene binary mixture. Yu and Li [2] reported the excess molar volumes of sulfolane in binary mixtures with six aromatic hydrocarbons (toluene, xylenes, ethylbenzene and 1,2,4-trimethylbenzene) at 298.15 K. Liquid–liquid equilibria in some binary and ternary mixtures with sulfolane have been studied by several researchers [3–7]. De Fre and Verhoeye [3] also reported the vapor–liquid equilibrium data of

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the systems sulfolane + benzene and sulfolane + toluene at 101.33 kPa, and a static method was used by Karvo [8] to measure the vapor pressures of these two systems. The vapor–liquid equilibria of the system sulfolane + water have been studied by Yu et al. [9]. However, for the mixtures of sulfolane with xylenes, ethylbenzene and 1,2,4-trimethylbenzene, vapor–liquid equilibrium data are not available in the literature.

When aromatics are separated from the mixture of sulfolane, the distillation at atmospheric pressure is generally used. Therefore, in this work we experimentally measured boiling points for the five binary mixtures of sulfolane with o-xylene, m-xylene, p-xylene, ethylbenzene and 1,2,4-trimethylbenzene. The data were treated thermodynamically, considering the non-ideality of both phases. The Margules [10], van Laar [10], Wilson [11], NRTL [12] and UNIQUAC [13] equations were fitted with the experimental boiling points of each binary mixture for their optimum parameters, and the compositions in the vapor-phase were calculated. A comparison of the five activity coefficient equations has been carried out.

2. Experimental

2.1. Materials

Sulfolane was supplied by Beijing Chemical Engineering Plant (chemical reagent) and was twice distilled at a pressure below 2.7 kPa. The purified sulfolane is colorless and odorless. Ethylbenzene, p-xylene, o-xylene and m-xylene are chemical reagents, and 1,2,4-trimethylbenzene is analytical reagent. These aromatic hydrocarbon compounds were purified by distillation and their middle fractions were collected. Then they were dried using activated type 0.5 nm molecular sieves, and the water content was found to be <0.01 mass% as determined by the Mitsubishi Moisture Meter (Model CA-05). To minimize the contact of these deliquescent reagents with moist air, the purified chemicals were kept in sealed bottles in desiccators. The normal boiling points at 101.33 kPa, refractive index values and densities at 298.15 K, of the purified chemicals were measured in this work and reported in Table 1 in comparison with the literature data [14–17] in order to demonstrate the purity of the compounds used in this work.

Table 1
Densities and refractive indexes at 298.15 K, and boiling points at 101.33 kPa, of the compounds

Compound	Density (g/cm^3)		Refractive index		Boiling point (K)	
	Experimental	Literature[a]	Experimental	Literature[a]	Experimental	Literature[a]
Sulfolane	1.26564[b]	1.26600[c]	1.4819[d]	1.4816[d]	560.47	560.50
o-Xylene	0.87567	0.87594	1.5029	1.50295	417.52	417.579
m-Xylene	0.85988	0.86009	1.4947	1.49464	412.25	412.270
p-Xylene	0.85648	0.85661	1.4932	1.49325	411.60	411.509
Ethylbenzene	0.86289	0.86253	1.4932	1.49320	409.39	409.343
1,2,4-Trimethylbenzene	0.87170	0.87174[e]	1.5024	1.50240[f]	442.45	442.53[f]

[a] The literature data were taken from Riddick et al. [14].

[b] Extrapolated value from experimental data.

[c] Interpolated data from Jannelli et al. [15].

[d] Data at 303.15 K.

[e] Data from Anderko [16].

[f] Data from Daubert and Danner [17].

2.2. Boiling point measurements

An inclined ebulliometer with a pump-like stirrer developed by Zhou et al. [18] and described by Yu et al. [19] was used. It is a recirculation type, in which both liquid and vapor phase recirculated continuously, and the equilibrium compositions of both phases can be determined. In the operation, about 80 cm^3 of solution is introduced into the ebulliometer. The mixture of liquid and vapor are carried from the boiling chamber upward to the equilibrium chamber, where the two phases after striking directly against the thermometer stem, separate into a liquid and vapor stream. Beneath the thermometer stem, there is a cup-like liquid-phase sampling chamber with a sampling port sealed by silicone rubber. The vapor is condensed in a condenser and flows to the mixing chamber. The liquid is cooled and passed through the liquid-phase sampling chamber to the mixing chamber, where it mixes with the condensate, returning to the boiling room for recirculation. When the equilibrium was reached, the liquid phase sample was taken from the liquid-phase sampling chamber by an injector. The thermometer used in the equilibrium chamber is a mercury thermometer. The uncertainty in the temperature measurement is ±0.05 K. The inclined ebulliometer was operated at atmospheric pressure. The atmospheric pressure measured by a mercury barometer is 101.33 ± 0.05 kPa in all boiling point measurements.

The compositions of the sampled liquid phase were analyzed by measuring their densities at 298.15 K with a vibrating tube densimeter, after calibration with gravimetrically prepared standard solutions. The accuracy of the equilibrium-composition measurements in mole fraction is ±0.0003. Since there is a large difference between the boiling points of sulfolane and of the aromatic hydrocarbons studied, the amount of the sulfolane present in the vapor phase was small. It is time consuming to reach steady state if the vapor phase sample is taken. Therefore, the vapor phase composition was not measured for the binary systems studied in the present work.

2.3. Density measurements

Densities of samples were measured at 298.15 K by an Anton Paar DMA60 vibrating tube density meter (Graz, Austria), in combination with one DMA602 remote cell. The temperature of the U-shaped tube was checked continuously using a calibrated digital thermometer (Anton Paar DT100-20) with an accuracy of ±0.01 K. The system was maintained at constant temperature to within ±0.005 K by a Hetotherm bath circulator (Heto, Type CB7). All the measurements were carried out at atmospheric pressure (101.33 kPa) and the pressure was measured by means of a mercury barometer.

The apparatus, the mixture standard sample preparation, and the procedure of the density measurements have been described elsewhere [20,21]. The excess volumes used for determination of sample composition of the five binary mixtures have been published in [2].

3. Results and discussion

The boiling points were measured at 101.33 kPa for the five binary systems of sulfolane with aromatic hydrocarbons. The results are shown in Table 2. The boiling points were correlated with

$$\hat{\phi}_i y_i p = \gamma_i x_i p_i^{\text{sat}} \phi_i^{\text{sat}} \exp\left[\frac{V_i^{\text{L}}(p - p_i^{\text{sat}})}{RT}\right], \quad i = 1, 2 \tag{1}$$

Table 2
Vapor–liquid equilibrium data for the binary system studied

T (K)	x_i	$y_1{}^a$	T (K)	x_i	$y_1{}^a$
Sulfolane (1) + *o*-xylene (2)					
419.16	0.0556	0.0045	435.69	0.6887	0.0252
421.08	0.1504	0.0087	436.92	0.7051	0.0264
422.13	0.2258	0.0108	440.30	0.7499	0.0310
424.05	0.2958	0.0124	443.28	0.7799	0.0353
425.12	0.3429	0.0134	447.53	0.8171	0.0430
430.66	0.5656	0.0189	452.01	0.8376	0.0492
432.07	0.6139	0.0208	468.49	0.9035	0.0915
Sulfolane (1) + *m*-xylene (2)					
412.81	0.0383	0.0035	426.06	0.6521	0.0166
413.94	0.0946	0.0061	429.37	0.7144	0.0197
415.07	0.1607	0.0078	432.83	0.7633	0.0237
416.86	0.2842	0.0096	435.09	0.7891	0.0267
420.13	0.4781	0.0122	441.92	0.8299	0.0338
421.16	0.5298	0.0131	445.89	0.8605	0.0427
423.13	0.5976	0.0147			
Sulfolane (1) + *p*-xylene (2)					
412.97	0.0541	0.0050	429.13	0.7566	0.0205
415.12	0.1958	0.0086	431.73	0.7854	0.0231
418.45	0.4618	0.0114	434.24	0.8021	0.0251
420.30	0.5619	0.0129	437.38	0.8322	0.0299
421.59	0.6121	0.0140	443.02	0.8550	0.0352
422.96	0.6799	0.0162	445.76	0.8706	0.0402
424.43	0.7068	0.0174	452.45	0.9021	0.0566
427.00	0.7461	0.0197			
Sulfolane (1) + ethylbenzene (2)					
411.23	0.0754	0.0043	421.90	0.6010	0.0140
411.94	0.1278	0.0058	422.82	0.6209	0.0146
413.47	0.2232	0.0075	426.30	0.6780	0.0170
414.67	0.2824	0.0083	432.96	0.7657	0.0238
416.27	0.3521	0.0093	435.56	0.7829	0.0259
417.64	0.4194	0.0102	441.93	0.8350	0.0358
419.64	0.5124	0.0118	447.51	0.8581	0.0433
420.67	0.5591	0.0128			
Sulfolane (1) + 1,2,4-trimethylbenzene (2)					
446.81	0.1584	0.0240	455.76	0.6238	0.0533
447.51	0.1980	0.0276	458.42	0.6871	0.0589
449.17	0.3042	0.0351	461.95	0.7479	0.0667
449.92	0.3557	0.0381	467.12	0.8054	0.0789
450.96	0.4125	0.0411	469.32	0.8252	0.0851
452.69	0.4964	0.0455	474.42	0.8582	0.0998
453.77	0.5480	0.0483	484.79	0.9213	0.1659
454.16	0.5609	0.0491			

[a] Calculated values from the Wilson equation.

Table 3
Coefficients used in the determination of the vapor pressure of the pure component

Component	T (K)	A	B	C	D
Sulfolane[a]	391.2–558.2	27.8073	4350.7	6.5633	
o-Xylene[b]	337.0–630.3	−7.53357	1.40968	−3.10985	−2.85992
m-Xylene[b]	332.0–617.0	−7.59222	1.39441	−3.22746	−2.40376
p-Xylene[b]	331.0–616.2	−7.63495	1.50724	−3.19678	−2.78710
Ethylbenzene[b]	330.0–617.1	−7.48645	1.45488	−3.37538	−2.23048
1,2,4-Trimethylbenzene[b]	358.0–649.1	−8.50002	2.98227	−6.02665	3.51307

[a] Coefficients [14] of the equation $\log(p^{sat}$ (kPa)$) = (A - B)/T$ (K) $- C \log T$ (K).

[b] Coefficients [22] of the equation $\ln(p^{sat}/p_c) = (1 - x)^{-1}(Ax + Bx^{1.5} + Cx^3 + Dx^6)$, where $x = 1 - T/T_c$.

where $\hat{\phi}_i$ and ϕ_i^{sat} are the fugacity coefficients of component i in the mixture and pure vapor, respectively. In Eq. (1), γ_i the activity coefficient of component i in the liquid phase, T the boiling points of the solution, p_i^{sat} the saturated vapor pressure of component i, V_i^L the saturated liquid molar volume of component i, x_i the liquid phase molar fraction and y_i the vapor phase molar fraction of component i.

The saturated vapor pressures of the pure components were obtained using the fitted parameters from Riddick et al. [14] and Reid et al. [22]. These are reproduced in Table 3. The liquid molar volumes were calculated from the Hankinson–Brobst–Thomson equation [22]. The fugacity coefficients were estimated by using the virial equation of state truncated after the second virial term. The second virial coefficients were obtained by using the method of Hayden and O'Connell [23]. The critical properties and other parameters required for estimating the second virial coefficients, obtained from Steele et al. [24], Domanska et al. [25], Prausnitz et al. [26] and Daubert and Danner [17], are listed in Table 4. The values of the association parameters for pure components and the solvation parameters for the binary mixtures studied were regarded as zero because the substances involved in the mixtures are non-associating fluids.

The experimental data of the five binary systems were correlated using the Margules, van Laar, Wilson, NRTL and UNIQUAC equations. The UNIQUAC pure component parameters for the surface area and volume of the molecules were obtained from a group contribution method [10] and are included in Table 4.

Table 4
Physical properties used in calculating the second virial coefficients, and UNIQUAC pure component parameters for volume and surface area of each component

Component	T_c (K)	p_c (MPa)	RD (nm)	μ (D)	r[a]	q[a]
Sulfolane	855.0[b]	7.290[b]	0.2910[c]	4.10[c]	4.036	3.206
o-Xylene[d]	630.37	3.733	0.3789	0.62	4.658	3.536
m-Xylene[d]	617.05	3.541	0.3897	0.40	4.658	3.536
p-Xylene[d]	616.26	3.511	0.3796	0.00	4.658	3.536
Ethylbenzene[d]	617.17	3.609	0.3821	0.58	4.597	3.508
1,2,4-Trimethylbenzene[e]	649.13	3.232	0.4199	0.30	5.393	4.104

[a] Data obtained from group contributions [10].

[b] Data from Steele et al. [24].

[c] Data from Domanska et al. [25].

[d] Data from Prausnitz et al. [26].

[e] Data from Daubert and Danner [17].

The binary parameters in the five activity coefficient equations were estimated based on minimization of the objective function F in terms of the calculated and experimental boiling point values. The function F used in this work can be expressed as

$$F = \left[\frac{1}{N} \sum_{j=1}^{N} \left(\frac{T_j^c - T_j^e}{T_j^e} \right)^2 \right]^{1/2} \tag{2}$$

The fitted parameters along with the average absolute deviations in the boiling point are listed in Table 5.

Table 5
Correlation parameters and absolute average deviations in boiling temperature for the binary systems studied

Model[a]	A_{12}[b]	A_{21}[b]	α_{12}	ΔT (%)
Sulfolane (1) + o-xylene (2)				
Margules	1.6347	1.3055		0.14
van Laar	1.6798	1.3117		0.13
Wilson	707.01	170.85		0.07
NRTL	380.95	597.39	0.593	0.06
UNIQUAC	2.01	215.90		0.16
Sulfolane (1) + m-xylene (2)				
Margules	1.8517	1.3703		0.20
van Laar	1.9301	1.3890		0.17
Wilson	870.78	158.91		0.10
NRTL	428.41	779.84	0.590	0.08
UNIQUAC	−33.63	286.10		0.19
Sulfolane (1) + p-xylene (2)				
Margules	1.9495	1.5031		0.17
van Laar	2.0147	1.5170		0.16
Wilson	950.53	197.45		0.15
NRTL	398.95	735.67	0.495	0.16
UNIQUAC	−3.03	261.19		0.18
Sulfolane (1) + ethylbenzene (2)				
Margules	1.7488	1.2501		0.15
van Laar	1.8207	1.2786		0.12
Wilson	794.03	122.74		0.08
NRTL	353.15	649.10	0.581	0.08
UNIQUAC	−44.38	282.91		0.14
Sulfolane (1) + 1,2,4-trimethylbenzene (2)				
Margules	1.5687	1.6244		0.25
van Laar	1.5663	1.6271		0.25
Wilson	656.68	349.33		0.18
NRTL	635.45	634.02	0.644	0.17
UNIQUAC	50.01	175.79		0.30

[a] All models are in $\ln \gamma$ form.
[b] Energy parameters $A_{ji} = (\lambda_{ji} - \lambda_{ii})/R$ for the Wilson equation, NRTL for the NRTL equation, and $A_{ji} = (U_{ji} - U_{ii})/R$ for the UNIQUAC model.
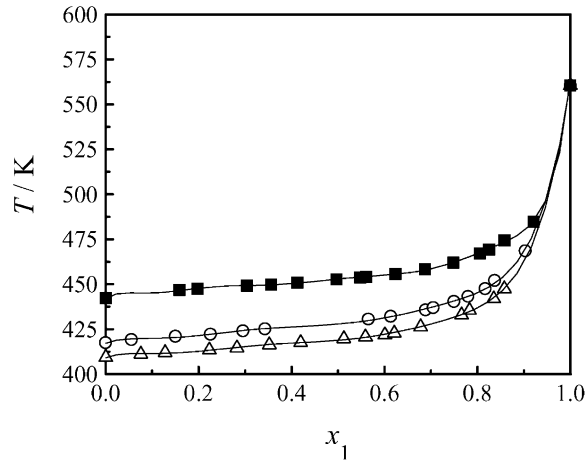
Fig. 1. Boiling points as a function of composition for binary mixtures of sulfolane with aromatic hydrocarbons. Symbols refer to the experimental data: (■) sulfolane + 1,2,4-trimethylbenzene; (○) sulfolane + ethylbenzene; (△) sulfolane + o-xylene; lines refer to the correlated results of Wilson equation.

The average absolute deviation in Table 5 is defined as

$$\Delta T = \frac{100}{N} \sum_{j=1}^{N} \left| \frac{T_j^{c} - T_j^{e}}{T_j^{e}} \right| \tag{3}$$

As seen from Table 5, the deviations in the boiling points are reasonably small, and this indicates that all five activity coefficient models are suitable to represent the binary experimental data. The NRTL and Wilson equations give the better results while the Margules and UNIQUAC equations exhibit the bigger deviations in boiling point for the five binary systems containing sulfolane. Considering the large differences in the boiling points of the binary mixtures, the deviations from all five activity coefficient models are within an acceptable range.

Fig. 1 gives the boiling points for mixtures of sulfolane with o-xylene, ethylbezene and 1,2,4-trimethyl-bezene. The T–x diagrams for the mixtures of sulfolane + m-xylene and sulfolane + p-xylene are similar to that of the mixture of sulfolane + ethylbenzene, and we did not included them in Fig. 1. As seen from Fig. 1, the experimental boiling points for the mixtures of sulfolane can be correlated by the Wilson equation with a good accuracy.

The activity coefficients of aromatic hydrocarbons and sulfolane in the five binary systems were predicted with the Wilson equation and the results are plotted in Figs. 2 and 3. The values of $\ln \gamma_i$ for the five binary systems are all positive. The activity coefficients of aromatic hydrocarbons in sulfolane solutions at infinite dilution are found from Fig. 2 to vary in the following order:

1, 2, 4-trimethylbenzene $>$ p-xylene $>$ m-xylene $>$ o-xylene $\approx$ ethylbenzene,

and from Fig. 3, one can see that the activity coefficient of sulfolane in different solutions at infinite dilution falls in the order:

p-xylene $>$ m-xylene $>$ ethylbenzene $>$ o-xylene $>$ 1, 2, 4-trimethylbenzene.
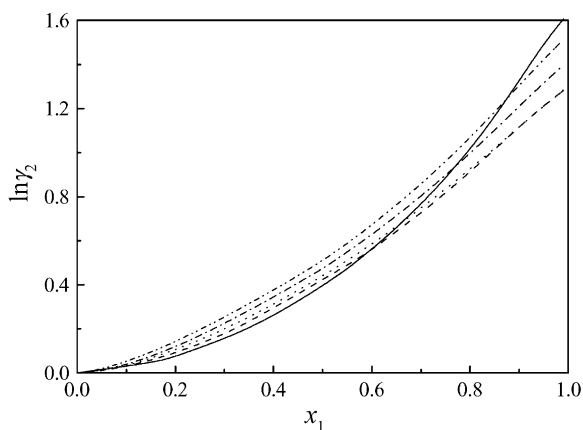
Fig. 2. Activity coefficients of aromatic hydrocarbons in sulfolane solutions. sulfolane (1) + *o*-xylene (2) (– – –) ; sulfolane (1) + *m*-xylene (2) (– · –); sulfolane (1) + *p*-xylene (2) (– · · –); sulfolane (1) + ethylbenzene (2) (· · ·) and sulfolane (1) + 1,2,4-trimethylbenzene (2) (—).

This order can be understood in term of the semi-empirical group-contribution schemes (UNIFAC) developed by Fredenslund et al. [27]. In the UNIFAC model, the activity coefficient is divided into two parts, the first a combinatorial term due to differences in the size and shapes of component molecules and the second, residual term, related directly to energy differences

$$\ln \gamma_i = \ln \gamma_i^{\mathrm{C}} + \ln \gamma_i^{\mathrm{R}} \tag{4}$$

Each molecule is divided into its component functional groups, and each pair of groups is assigned a value of the group interaction parameter. If Eq. (4) is applied to the system sulfolane + xylenes, the activity coefficient should be the same in the three systems of xylenes because the component functional groups and their numbers are identical in these systems. However, the activity coefficients from the experiment are different in these systems of xylenes. Actually, the dipole–dipole interaction contribution between
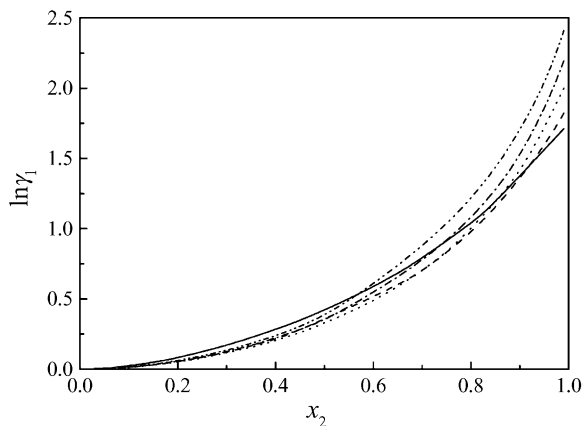

Fig. 3. Activity coefficients of sulfolane in different solutions. The lines have the same meaning as in Fig. 2.

molecules is not included in the original UNIFAC. Therefore, the dipole–dipole interaction contribution should be added to the activity coefficient.

$$\ln \gamma_i = \ln \gamma_i^C + \ln \gamma_i^R + \ln \gamma_i^D \tag{5}$$

For the systems containing sulfolane, the bigger the value of the dipole moment of the aromatic hydrocarbon, the smaller the activity coefficient in the solution. From the data of dipole moment in Table 4, one can conclude that the activity coefficient in the solution of sulfolane falls in the order p-xylene $> m$-xylene $> o$-xylene. In the system, sulfolane $+$ ethylbenzene, the main groups and their number are identical with the system of xylene, and the residual term of the activity coefficient can be approximately regarded as having the same value. The difference in the activity coefficient between the systems of ethylbenzene and xylene comes from the combinatorial term and dipole–dipole interaction. The combinatorial term can be obtained from the UNIQUAC equation and its value for the system of ethylbenzene is slightly smaller than that for the system of xylene. However, Table 4 shows that the dipole moment of ethylbenzene is smaller than that of o-xylene and bigger than that of m-xylene. Therefore, the activity coefficient in the solution of ethylbenzene should be equal or slightly bigger than that in the solution of o-xylene and smaller than that in the solutions of m-xylene. From the experimental data, we can see that introduction of a methyl group in the ring of benzene will increase the activity coefficient of aromatic hydrocarbon and decrease the activity coefficient of sulfolane in the system sulfolane $+$ aromatic hydrocarbon at infinite dilution. This trend is the same as that in the system tetraethylene glycol $+$ aromatic hydrocarbon [19].

4. Conclusions

Experimental boiling points for the five binary systems of sulfolane $+ o$-xylene, sulfolane $+ m$-xylene, sulfolane $+ p$-xylene, sulfolane $+$ ethylbenzene, and sulfolane $+$ 1,2,4-trimethylbenzene were determined at atmospheric pressure. Analysis of the experimental data for the five binary systems by using the Margules, van Laar, Wilson, NRTL and UNIQUAC equations shows that all five equations generally give acceptable results. Among the five activity coefficient models, the Wilson and NRTL equations can best represent the experimental boiling points for the binary systems studied, and the Margules and NRTL equations give bigger deviations. The activity coefficients of the components in the liquid phase were qualitatively explained by using the UNIFAC model with the consideration of dipole–dipole interactions. The order of the activity coefficients in different solutions at infinite dilution is accord with the prediction from the UNIFAC model by including the dipole–dipole interactions.

List of symbols

A_{12}, A_{21}	parameters of tvhe liquid activity coefficient model
A, B, C, D	constants of the vapor-pressure equation
F	objective function
g	parameter of NRTL equation (J/mol)
N	number of experimental points
p	pressure (kPa)
q	UNIQUAC area parameter
r	UNIQUAC volume parameter
RD	mean radius of gyration (nm)

R	universal gas constant (8.3144 J/mol K)
T	boiling point (K)
U	energy parameter of UNIQUAC equation (J/mol)
V	molar volume (cm^3/mol)
x	mole fraction in the liquid phase
y	mole fraction in the vapor phase

Greek letters

α	parameter of NRTL equation
γ	liquid-phase activity coefficient
ΔT	absolute average deviation in boiling point (%)
ϕ	vapor-phase fugacity coefficient in pure component
$\hat{\phi}$	vapor-phase fugacity coefficient in mixture
λ	energy parameter of Wilson equation (J/mol)
μ	dipole moment (D)

Superscripts

c	calculated value
C	combinatorial term
D	dipole–dipole interaction term
e	experimental value
L	liquid state
R	residual term
sat	saturated state

Subscripts

| c | critical state |
| 1,2,i | component 1, 2, i |

Acknowledgements

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