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Vapor-liquid equilibra for cyclohexane+2-propoxyethanol and cyclohexane+2-butoxyethanol systems

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Abstract−2-propoxyethanol (C_3E_1) and 2-butoxyethanol (C_4E_1) are nonionic surfactants which are a particularly interesting class of substances due to both inter-molecular and intra-molecular association, related to the presence of O and OH in the same molecule. Binary (vapor+liquid) equilibrium data were measured for cyclohexane+2-propoxyethanol and cyclohexane+2-butoxy ethanol systems at temperatures ranging from 303.15 K to 323.15 K at 10 K intervals. A static apparatus was used in this study. Two systems show positive deviation from Raoult's law and no azeotrope. The experimental data were correlated well with Peng-Robinson-Stryjek-Vera equation of state using Wong-Sandler mixing rule incorporating the non-random-two-liquid model.

Key words: Surfactants, 2-Propoxyethanol, 2-Butoxyethanol, Cyclohexane, Association

INTRODUCTION

Surfactant molecules constitute an important class of chemicals with numerous applications in chemical process industries, in the formulation of agricultural chemicals, pharmaceutical and cosmetic products, in mineral processing technologies and in food processing industries. The wide-spread applications of surfactants have their origin in their intriguing molecular characteristics, namely, they are composed of a polar part that likes water and a non-polar part that dislikes water but is compatible with oil. This dual nature endows the surfactants with interfacial and thermodynamic properties. It is very important to accurately predict and calculate phase behavior for mixtures containing surfactant systems in the design and operation of separation processes. Mixtures containing ethoxylated alcohol [$H\text{-}(CH_2)_i(OCH_2CH_2)_jOH$ or C_iE_j] surfactants have been attracting considerable attention due to intra and inter-molecular interaction, related to the presence of O or OH groups, or both, in the same or different molecules. Phase equilibrium data have been extensively measured [1-4] for surfactants and water systems, but limited data are reported for surfactants and oils systems (e.g. C_1E_1+n-hexane, n-heptane, cyclohexane [5] and C_2E_1+n-hexane, n-heptane, cyclohexane [6]). Phase equilibrium data for surfactants and hydrocarbons systems are essential in the design and operation of separation processes for mixtures containing surfactant systems [7-10].

In an earlier study, we measured isothermal vapor-liquid equilibrium (VLE) data of C_3E_1+n-hexane, n-heptane [7], C_4E_1+n-hexane, n-heptane [8] and isobaric VLE data of iC_3E_1+n-hexane, n-heptane [9], C_3E_1+n-heptane, n-octane [10]. In this study, we measured isothermal VLE data for cyclohexane+2-propoxyethanol (C_3E_1) and cyclohexane+2-butoxyethanol (C_4E_1) systems at four different temperatures ranging from 303.15 to 343.15 K. The experimental data were correlated with Peng-Robinson-Stryjek-Vera equation of state [11] using van der Waals one-fluid mixing rule and Wong-Sandler mixing rule [12].

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EXPERIMENTAL SECTION

1. Materials

2-Propoxyethanol (C_3E_1, molecular weight 104.15, CAS No. 2807-30-9, $C_5H_{12}O_2$) and 2-butoxyethanol (C_4E_1, molecular weight 118.17, CAS No. 111-76-2, $C_6H_{14}O_2$) were obtained from Sigma-Aldrich Corporation at purity of 99.4% and Tokyo Kasei Chemical Corporation at purity of 99.0%, respectively, and their chemical structure. Cyclohexane was supplied by Sigma-Aldrich Corporation at purities of 99.5%. All materials were used directly from the manufacturers without further purification.

2. Experimental Apparatus and Procedure

Experimental details are explained in previous papers [7,8], similar to Gibbs and van Ness [13] and Giles et al. [14], in which total pressure is measured as a function of overall composition in the equilibrium cell. Pressure, temperature, and liquid-phase composition are measured by experiments, and vapor phase composition is calculated by using the measured pTx data. Fig. 1 shows a schematic diagram of the experimental apparatus. The system should be settled to reach equilibrium until the pressure oscillates within ±0.01 kPa but does not require analysis of vapor and liquid composition. This apparatus was designed to operate up to 423 K. The equilibrium cell was made of 316 stainless-steel, and its internal volume was approximately 100 mL. The equilibrium temperature of the system was measured by a 100 Ω platinum resistance thermometer (PRT), model 5614 by Hart Scientific Co., with an accuracy of ±0.006 K, and a 1502A digital indicator by Hart Scientific Co. The PRT was calibrated by comparison to a standard platinum

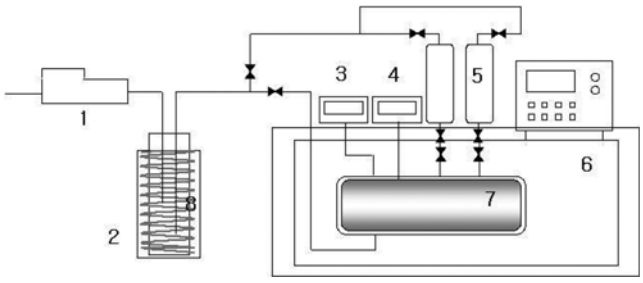

Fig. 1. Schematic diagram of the experimental apparatus.

1. Vacuum pump
2. Trap
3. Temperature indicator
4. Pressure indicator
5. Reservoir
6. Isothermal bath
7. Equilibrium cell
8. Cooling coil

resistance thermometer (SPRT). The SPRT is calibrated to the international temperature scale of 1990 (ITS-90). A model Super TJE (<50 psi) pressure transducer by Sensotec Co. was connected to a model L20010WM1 digital indicator by Laurel and used to measure the pressure of the system. The estimated accuracy of the digital pressure gauge is ±0.01 kPa. A digital balance, MP-3000 by CHYO, with a minimum accuracy of ±0.001 g, was used to measure liquid sample weight.

Experimental procedures are briefly described as follows. First, the samples were injected into each cylinder and the cylinders were degassed. The degassed cylinders were weighed by a digital balance separately. Then, the cylinders were attached to the equilibrium cell and the whole system containing the cell was sufficiently evacuated to remove all impurities. After checking for leaks, we charged the surfactant into the cell, and then injected cyclohexane. Liquid phase volume was checked through the cell window. After charging a proper amount of samples, we removed the sample cylinders from the cell and measured the weight of the cylinders in order to calculate the charged composition in the cell. When equilibrium was reached at the desired temperature, the pressure was measured.

CORRELATIONS

Experimental data were correlated with the Peng-Robinson-Stryjek-Vera equation of state (PRSV EOS) [11] using van der Waals one-fluid mixing rule and Wong-Sandler mixing rule [12]. The PR SV EOS is expressed as follows:

$$P = \frac{RT}{V-b} - \frac{a(T)}{V(V+b)+b(V-b)} \tag{1}$$

$$a(T) = 0.45724\frac{R^2T_c^2}{P_c}\alpha(T) \tag{2}$$

$$b = 0.07780\frac{RT_c}{P_c} \tag{3}$$

$$\alpha(T) = [1+\kappa(1-T_r^{0.5})+\kappa_1(1-T_r)(0.7-T_r)]^2 \tag{4}$$

$$\kappa = 0.378893 + 1.4897153\,\omega - 0.17131848\,\omega^2 + 0.0196554\,\omega^3 \tag{5}$$

where T_c is the critical temperature, P_c is the critical pressure, T_r is the reduced temperature, ω is the acentric factor and κ_1 is pure compound adjustable parameter [10].

The van der Waals one-fluid mixing rule and combining rule are represented as follows:

$$a_m = \sum\sum x_i x_j a_{ij} \tag{6}$$

$$b_m = \sum x_i b_i \tag{7}$$

$$a_{ij} = \sqrt{a_i a_j}(1-k_{ij}) \tag{8}$$

where k_{ij} is binary interaction parameter.

The Wong-Sandler mixing rule and combining rule are represented as follows:

$$b_m = \frac{\sum_i\sum_j x_i x_j\left(b-\frac{a}{RT}\right)_{ij}}{1-\sum_i x_i\frac{a_i}{b_i RT}-\frac{A_\infty^E}{CRT}} \tag{9}$$

$$a_m = b_m\left(\sum_i x_i\frac{a_i}{b_i}+\frac{A_\infty^E}{C}\right) \tag{10}$$

$$\left(b-\frac{a}{RT}\right)_{ij} = \frac{\left(b_i-\frac{a_i}{RT}\right)+\left(b_j-\frac{a_j}{RT}\right)}{2}(1-k_{ij}) \tag{11}$$

where $C=\ln(\sqrt{2}-1)/\sqrt{2}$ for the PRSV EOS. Because the excess Helmholtz free energy of mixing at infinite pressure is assumed to equal the excess Gibbs free energy (G^E) at low pressure, the G^E model is used in place of A_∞^E. We used the non-random-two-liquid (NRTL) model [15] as a G^E model in this study:

$$\frac{G^E}{RT} = \sum_{i=1} x_i\frac{\sum_{j=1} x_j G_{ji}\tau_{ji}}{\sum_{k=1} x_k G_{kj}} \tag{12}$$

$$\frac{G_{ij}}{RT} = \exp(-\alpha_{ij}\tau_{ij}),\quad \alpha_{ij} = \alpha_{ji} \tag{13}$$

where τ_{ij} and τ_{ji} are the interaction parameters and α_{ij} is the non-randomness parameter. We fixed the non-randomness parameter α_{ij} to 0.3 in this study.

The three interaction parameters (k_{ij}, τ_{ij}, τ_{ji}) were obtained from the Marquardt algorithm which was used to minimize the following objective function, the absolute average deviations for pressure (AADP):

$$AADP = \frac{1}{N_{exp}}\sum_i^{N_{exp}}\left|\frac{p_{exp}-p_{cal}}{p_{exp}}\right| \tag{14}$$

where N_{exp} is the number of experimental data points and p_{exp} and p_{cal} are the experimental and the calculated pressures, respectively.

RESULTS AND DISCUSSION

The vapor pressures of the pure components (cyclohexane, C_3E_1, C_4E_1) measured are reported in Table 1 and compared with smoothed values obtained from the Dortmund Data Bank (DDB) [16]. Good agreement was shown between them. Pure parameters for the PRSV EOS are listed in Table 2 and κ_1 is fitted to vapor pressure data using our experimental and DDB. To test the two mixing rules of PRSV

Table 1. Comparison of pure component vapor pressures

	T=303.15 K		T=313.15 K		T=323.15 K		T=333.15 K	
	Exp	Lit. [15]	Exp	Lit. [15]	Exp	Lit. [15]	Exp	Lit. [15]
	p_{vap}/kPa							
Cyclohexane	16.22	16.23	24.56	24.58	36.21	36.18	52.19	52.21
C_3E_1	0.45	0.44	0.89	0.89	1.59	1.58	2.77	2.79
C_4E_1	0.18	0.19	0.37	0.38	0.72	0.72	1.31	1.29

Table 2. Pure parameters for the PRSV EOS

PRSV EOS	T_c (K)	P_c (MPa)	ω	κ_1
Cyclohexane	553.58	4.073	0.210	0.0452
C_3E_1	615.20	3.651	0.486	0.0754
C_4E_1	633.90	3.270	0.521	0.0206

EOS in surfactants and hydrocarbons systems, VLE experimental data for cyclohexane+C_1E_1 and cyclohexane+C_2E_1 system were correlated with the PRSV EOS with two mixing rules and reported in Table 3.

As shown in Figs. 2 and 3, the Wong-Sandler mixing rule can predict the azeotropic behavior and shows better calculated results than van der Waals mixing rule. Therefore, PRSV EOS with Wong-Sandler mixing rule is a more acceptable model in surfactants and cyclohexane systems.

In this study, the pTx measurements were measured at temperature ranging from 303.15 K to 333.15 K for cyclohexane+C_3E_1 and cyclohexane+C_4E_1 systems and are listed in Tables 4 and 5, respectively. Experimental uncertainty of pressure, σ_p is within 0.016 kPa. Correlated results and binary interaction parameters with the PRSV

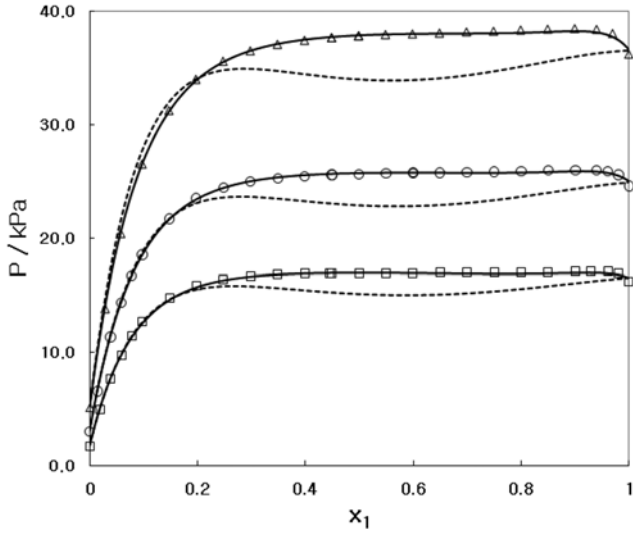

Fig. 2. Vapor-liquid equilibria of the cyclohexane (1)+C_1E_1 (2) system: experimental data at (□) 303.15 K, (○) 313.15 K, (△) 323.15 K; dotted lines: PRSV EOS with vdW mixing rule, solid lines: PRSV EOS with Wong-Sandler mixing rule.

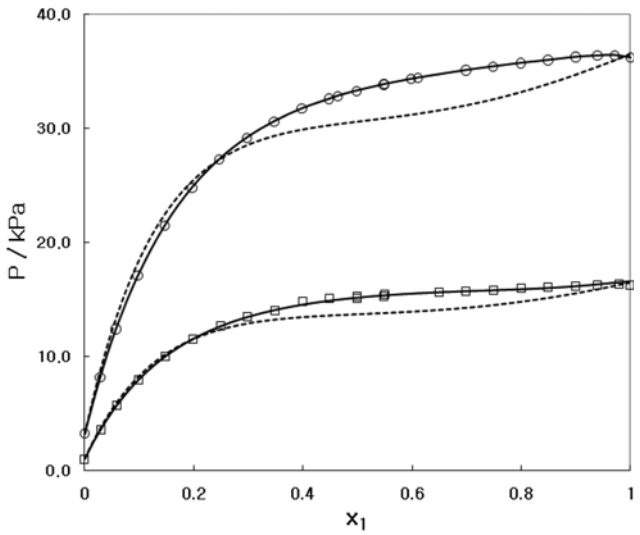

Fig. 3. Vapor-liquid equilibria of the cyclohexane (1)+C_2E_1 (2) system: experimental data at (□) 303.15 K, (○) 323.15 K; dotted lines: PRSV EOS with vdW mixing rule, solid lines: PRSV EOS with Wong-Sandler mixing rule.

Table 3. Binary parameters and the average absolute deviations of the PRSV EOS with 2 mixing rule for cyclohexane+C_1E_1, cyclohexane+C_2E_1 systems

Systems	T/K	PRSV with vdW		PRSV with WS			
		k_{12}	AADP[a]	k_{12}	τ_{12}	τ_{21}	AADP[a]
Cyclohexane	303.15	0.091	0.060	−0.120	3.148	2.028	0.004
+C_1E_1	313.15	0.096	0.066	−0.110	3.140	1.960	0.003
	323.15	0.101	0.069	−0.106	3.149	1.893	0.003
Cyclohexane	303.15	0.081	0.060	−0.167	3.100	1.238	0.004
+C_2E_1	323.15	0.089	0.059	−0.180	3.122	1.209	0.002

[a] $\mathrm{AADP}=\frac{1}{N_{exp}}\sum_{i}^{N_{cal}}\left|\frac{p_{exp}-p_{cal}}{p_{exp}}\right|$

Table 4. (Vapor-liquid) equilibrium measurements for the cyclohexane (1)+C_3E_1 (2) system

T/K	$x_{1,exp}$	P_{exp}/kPa	σ_P/kPa	P_{cal}/kPa	ΔP^a
303.15	0.051	3.46	0.011	3.48	0.007
	0.103	5.92	0.008	5.92	0.000
	0.142	7.41	0.009	7.42	0.001
	0.213	9.78	0.008	9.60	0.018
	0.292	11.60	0.005	11.42	0.015
	0.361	12.74	0.009	12.63	0.009
	0.441	13.70	0.013	13.70	0.000
	0.481	13.95	0.005	14.13	0.013
	0.550	14.72	0.007	14.73	0.001
	0.613	15.01	0.014	15.15	0.009
	0.691	15.28	0.007	15.50	0.015
	0.750	15.69	0.008	15.68	0.001
	0.812	15.84	0.004	15.80	0.002
	0.863	15.90	0.011	15.89	0.001
	0.921	16.13	0.007	16.04	0.006
	0.962	16.17	0.008	16.24	0.004
313.15	0.051	5.43	0.013	5.43	0.000
	0.103	9.05	0.011	9.08	0.004
	0.142	11.37	0.009	11.32	0.005
	0.213	14.45	0.007	14.54	0.006
	0.292	17.35	0.012	17.19	0.009
	0.361	18.86	0.010	18.92	0.003
	0.441	20.52	0.008	20.43	0.004
	0.481	21.01	0.012	21.03	0.001
	0.550	21.95	0.008	21.87	0.004
	0.613	22.18	0.007	22.46	0.012
	0.691	22.92	0.009	22.99	0.003
	0.750	23.29	0.011	23.29	0.000
	0.812	23.59	0.007	23.55	0.002
	0.863	23.86	0.006	23.77	0.004
	0.921	24.18	0.015	24.11	0.003
	0.962	24.34	0.012	24.48	0.006
323.15	0.051	8.08	0.012	8.18	0.012
	0.103	13.36	0.014	13.46	0.008
	0.142	16.83	0.008	16.69	0.008
	0.213	21.56	0.007	21.35	0.010
	0.292	25.14	0.014	25.14	0.000
	0.361	27.61	0.013	27.61	0.000
	0.441	29.81	0.009	29.76	0.002
	0.481	30.69	0.007	30.61	0.003
	0.550	31.81	0.016	31.81	0.000
	0.613	32.40	0.008	32.66	0.008
	0.691	33.27	0.013	33.46	0.006
	0.750	34.02	0.012	33.93	0.003
	0.812	34.21	0.008	34.38	0.005
	0.863	34.60	0.007	34.77	0.005
	0.921	35.48	0.014	35.34	0.004
	0.962	35.96	0.011	35.90	0.002
333.15	0.051	12.14	0.012	12.13	0.001
	0.103	19.53	0.013	19.61	0.004
	0.142	24.16	0.009	24.16	0.000
	0.213	30.60	0.012	30.66	0.002
	0.292	36.08	0.008	35.90	0.005
	0.361	39.32	0.013	39.27	0.001
	0.441	42.19	0.016	42.18	0.000
	0.481	43.01	0.013	43.33	0.007
	0.550	44.95	0.009	44.95	0.000
	0.613	46.12	0.008	46.12	0.000
	0.691	47.15	0.016	47.28	0.003
	0.750	47.86	0.014	48.02	0.003
	0.812	48.43	0.012	48.77	0.007
	0.863	49.35	0.008	49.45	0.002
	0.921	50.57	0.016	50.40	0.003
	0.962	51.53	0.011	51.25	0.006

$^a\Delta P = |P_{i,cal} - P_{i,exp}|/P_{i,exp}$

Table 5. (Vapor-liquid) equilibrium measurements for the cyclohexane (1)+C_4E_1 (2) system

T/K	$x_{1,exp}$	P_{exp}/kPa	σ_P/kPa	P_{cal}/kPa	ΔP^a
303.15	0.048	2.69	0.011	2.71	0.007
	0.097	4.84	0.008	4.84	0.000
	0.138	6.39	0.006	6.35	0.006
	0.205	8.30	0.012	8.40	0.013
	0.289	10.42	0.010	10.41	0.001
	0.358	11.75	0.009	11.71	0.003
	0.437	13.04	0.005	12.91	0.010
	0.492	13.63	0.004	13.59	0.003
	0.549	14.04	0.016	14.18	0.010
	0.622	14.54	0.016	14.78	0.017
	0.702	15.10	0.013	15.26	0.011
	0.748	15.49	0.009	15.46	0.002
	0.807	15.64	0.011	15.64	0.000
	0.851	15.74	0.013	15.74	0.000
	0.917	15.91	0.010	15.91	0.000
	0.959	16.12	0.009	16.12	0.000
313.15	0.048	4.23	0.010	4.19	0.008
	0.097	7.44	0.011	7.40	0.005
	0.138	9.58	0.016	9.66	0.009
	0.205	12.70	0.009	12.70	0.000
	0.289	15.61	0.007	15.64	0.002
	0.358	17.78	0.011	17.52	0.006
	0.437	19.17	0.011	19.24	0.004
	0.492	20.20	0.013	20.21	0.001
	0.549	21.01	0.008	21.06	0.003
	0.622	22.18	0.014	21.94	0.011
	0.702	22.67	0.011	22.67	0.000
	0.748	22.67	0.013	22.99	0.003
	0.807	23.24	0.010	23.32	0.004
	0.851	23.71	0.009	23.54	0.007
	0.917	24.11	0.016	23.91	0.008
	0.959	24.30	0.013	24.30	0.000
323.15	0.048	6.30	0.010	6.29	0.002
	0.097	10.97	0.011	10.97	0.000
	0.138	14.18	0.008	14.26	0.005
	0.205	18.60	0.009	18.65	0.003

Table 5. Continued

T/K	$x_{1,exp}$	P_{exp}/kPa	σ_P/kPa	P_{cal}/kPa	ΔP^a
323.15	0.289	23.07	0.013	22.86	0.009
	0.358	25.51	0.007	25.53	0.001
	0.437	27.88	0.005	27.94	0.002
	0.492	29.30	0.013	29.30	0.000
	0.549	30.50	0.014	30.48	0.001
	0.622	31.65	0.009	31.72	0.002
	0.702	32.54	0.008	32.77	0.007
	0.748	33.28	0.012	33.27	0.000
	0.807	33.86	0.016	33.82	0.001
	0.851	34.23	0.013	34.22	0.000
	0.917	35.09	0.009	34.92	0.005
	0.959	35.43	0.012	35.58	0.004
333.15	0.048	9.25	0.011	9.20	0.005
	0.097	15.84	0.014	15.84	0.000
	0.138	20.34	0.011	20.48	0.007
	0.205	26.92	0.016	26.64	0.011
	0.289	32.20	0.009	32.48	0.009
	0.358	36.02	0.012	36.14	0.003
	0.437	39.83	0.013	39.42	0.010
	0.492	41.15	0.008	41.26	0.003
	0.549	42.88	0.007	42.86	0.000
	0.622	44.31	0.012	44.56	0.006
	0.702	46.08	0.016	46.07	0.000
	0.748	46.83	0.015	46.83	0.000
	0.807	48.00	0.008	47.74	0.005
	0.851	48.40	0.007	48.43	0.001
	0.917	49.76	0.011	49.67	0.002
	0.959	50.86	0.012	50.73	0.003

$^a\Delta P = |P_{i,cal} - P_{i,exp}|/P_{i,exp}$

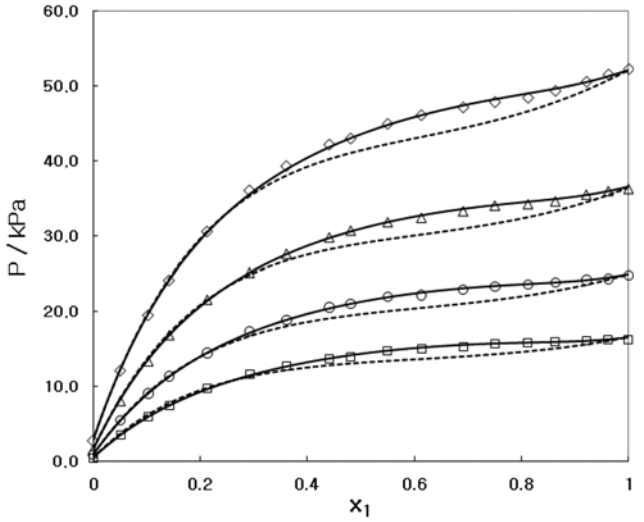

Fig. 4. Vapor-liquid equilibria of the cyclohexane (1)+C_3E_1 (2) system: experimental data at (□) 303.15 K, (○) 313.15 K, (△) 323.15 K, (◇) 333.15 K; dotted lines: PRSV EOS with vdW mixing rule, solid lines: PRSV EOS with Wong-Sandler mixing rule.

EOS with van der Waals and Wong-Sandler mixing rule are summarized in Table 6. The PRSV EOS with Wong-Sandler mixing rule correlated within 0.006, 0.005 AADP for cyclohexane+C_3E_1 and cyclohexane+C_4E_1 systems, respectively, while the PRSV EOS with van der Waals mixing rule correlated within 0.060, 0.046 AADP for two system, respectively. Figs. 4 and 5 show the deviations between the measured data and the calculated values of pressure for cyclohexane+C_3E_1 and cyclohexane+C_4E_1 systems, respectively. As shown in these figures, the PRSV EOS with Wong-Sandler mixing rule shows better calculated results than that with van der Waals mixing rule. Note that the two systems show positive deviation from Raoult's law and no azeotrope. As length of hydrocarbon tails is large, two phase regions are small and solubilities increase, as shown in Figs. 2, 3, 4 and 5.

CONCLUSION

We measured binary (vapor+liquid) equilibrium data for two systems, cyclohexane+C_3E_1 and cyclohexane+C_4E_1, at temperature ranging from 303.15 K to 333.15 K at 10 K intervals. The PRSV EOS with van der Waals mixing rule and Wong-Sandler mixing rule involving NRTL model was used to fit the experimental data. The PRSV EOS with Wong-Sandler mixing rule shows better cal-

Table 6. Binary parameters and the average absolute deviations of the PRSV EOS with 2 mixing rule for cyclohexane+C_3E_1, cyclohexane+C_4E_1 systems

Systems	T/K	PRSV with vdW		PRSV with WS			
		k_{12}	AADP^a	k_{12}	τ_{12}	τ_{21}	AADP^a
Cyclohexane	303.15	0.078	0.060	−0.226	3.922	0.917	0.006
+C_3E_1	313.15	0.079	0.048	−0.201	3.586	0.888	0.004
	323.15	0.085	0.041	−0.179	3.416	0.861	0.005
	333.15	0.088	0.031	−0.147	3.156	0.835	0.003
Cyclohexane	303.15	0.070	0.046	−0.227	4.265	0.778	0.005
+C_4E_1	313.15	0.074	0.038	−0.225	4.057	0.798	0.004
	323.15	0.080	0.030	−0.204	3.807	0.774	0.003
	333.15	0.083	0.024	−0.180	3.527	0.751	0.004

$^a AADP = \frac{1}{N_{exp}} \sum_{i}^{N_{exp}} \left| \frac{p_{exp} - p_{cal}}{p_{exp}} \right|$

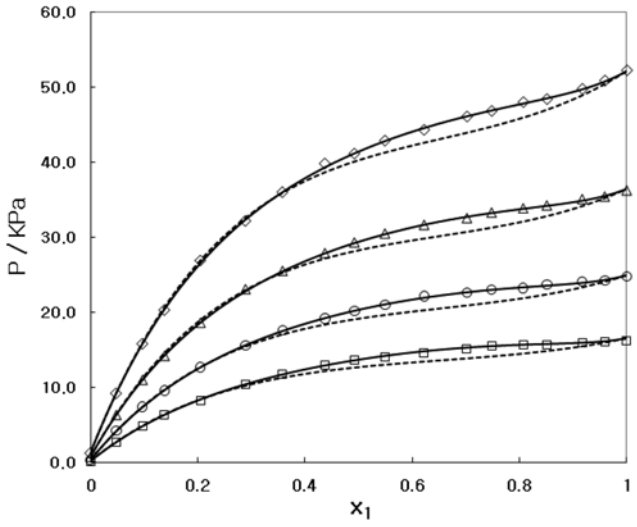

Fig. 5. Vapor-liquid equilibria of the cyclohexane (1)+C_4E_1 (2) system: experimental data (□) 303.15 K, (○) 313.15 K, (△) 323.15 K, (◇) 333.15 K; dotted lines: PRSV EOS with vdW mixing rule, solid lines: PRSV EOS with Wong-Sandler mixing rule.

culated results than that with van der Waals mixing rule. Correlation results of PRSV EOS with Wong-Sandler mixing rule are in good agreement within 0.006, 0.005 AADP for cyclohexane+C_3E_1 and cyclohexane+C_4E_1 systems, respectively. The two systems show no azeotrope and positive deviation from Raoult's law.

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