

Vapor–Liquid Equilibria of Cyclohexanol with Carbon Dioxide, Ethane, or Nitrogen at Elevated Pressures

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Vapor–liquid equilibrium (VLE) data were measured for the binary systems of cyclohexanol with carbon dioxide, ethane, or nitrogen at temperatures from 333.15 K to 453.15 K and pressures up to 190 bar. The saturated vapor compositions were correlated with the density of the light components. Henry's constants of the gases dissolved in cyclohexanol were calculated with the aid of the Krichevsky–Ilinskaya equation. The new VLE data were also correlated by the Peng–Robinson and the Patel–Teja equations of state. In general, the Patel–Teja equation incorporating the one-fluid, two-parameter van der Waals mixing rule yielded the best representation.

Introduction

Phase-equilibrium data at elevated pressures are of interest for model developments and engineering applications. Systems containing a supercritical component are receiving much attention. Recently, Dohrn et al. (1995) made a comprehensive review on high-pressure vapor–liquid equilibrium (VLE) measurements. In the past years, we have focused the VLE measurements on the binary systems of an alkanol with a supercritical fluid (Weng and Lee, 1992a; Lee and Chen, 1994; Weng et al., 1994). Cyclohexanol was selected in the present study as a model compound of the cyclic alcohols. The phase-equilibrium compositions were measured for three binary mixtures of cyclohexanol with carbon dioxide, ethane, or nitrogen at temperatures from (333.15 to 453.15) K and in a pressure range of (10 to 190) bar. Among these three binary systems, no existing data were found for cyclohexanol + ethane. The solubilities of carbon dioxide in cyclohexanol were measured by Begley et al. (1965) at near ambient conditions. Meanwhile, Lebedeva and Grokholskaya (1971) investigated the solubilities of nitrogen in cyclohexanol and the saturated vapor compositions of cyclohexanol + nitrogen at temperatures from (373.15 to 473.15) K and pressures up to 140 bar.

Experimental Work

The semiflow VLE apparatus used in this work is the same as that used in our previous studies (Lee and Chen, 1994; Weng et al., 1994). A detailed description of the equipment and the operation has been given elsewhere (Lee and Chao, 1988; Weng and Lee, 1992b). A silicon-oil thermostated bath controlled the equilibrium cell within ± 0.03 K. The cell's pressure was monitored by a pressure transducer (model PDCR-330, (0 to 350) bar, accurate to $\pm 0.1\%$, Druck Limited). The fluctuation of the cell's pressure was regulated within ± 0.1 bar in the course of the sampling. A microthermometer (model 1506, accurate to ± 0.02 K, Hart Scientific) with a platinum RTD sensor measured the equilibrium temperature. At least four samples were taken at each equilibrium condition and averaged. In general, the phase compositions were reproduced to $\pm 2\%$. However, the uncertainty could be $\pm 10\%$ for the mole fraction of the dilute component as low as 10^{-4} .

Carbon dioxide (99.8+%), ethane (99+%, CP grade), and nitrogen (99.9+%) were supplied by San-Fu Chemical Co.

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Table 1. Phase Compositions for Cyclohexanol (1) + Carbon Dioxide (2)

<i>P</i> /bar	<i>y</i> ₁	<i>x</i> ₁	<i>P</i> /bar	<i>y</i> ₁	<i>x</i> ₁
<i>T</i> = 333.15 K					
10.0	0.00155	0.9655	90.0	0.00370	0.692
20.0	0.000776	0.9314	110.0	0.00841	0.625
30.0	0.000471	0.897	130.0	0.0174	0.564
40.0	0.000481	0.865	150.0	0.0306	0.498
55.0	0.000663	0.815	170.0	0.0468	0.429
70.0	0.00161	0.761	190.0	0.0651	0.363
<i>T</i> = 393.15 K					
10.0	0.0267	0.9741	100.0	0.0115	0.714
20.0	0.0149	0.9464	120.0	0.0143	0.655
30.0	0.0112	0.9204	140.0	0.0187	0.601
40.0	0.00947	0.892	165.0	0.0277	0.528
60.0	0.00835	0.836	190.0	0.0424	0.441
80.0	0.00921	0.773			
<i>T</i> = 453.15 K					
10.0	0.156	0.9806	100.0	0.0479	0.748
20.0	0.0896	0.9563	120.0	0.0497	0.694
30.0	0.0679	0.9328	140.0	0.0543	0.634
45.0	0.0503	0.894	165.0	0.0629	0.569
60.0	0.0474	0.857	190.0	0.0791	0.490
80.0	0.0459	0.800			

(Taiwan). Cyclohexanol (99+%) was purchased from Aldrich Chemicals (USA). No further purification of those chemicals was made.

The equilibrium phase compositions of cyclohexanol + carbon dioxide, cyclohexanol + ethane, and cyclohexanol + nitrogen are compiled in Tables 1–3, respectively. Figures 1–3 present the respective pressure–composition diagrams for those investigated systems (open symbols). The left portion of the graphs illustrates the gas solubilities in cyclohexanol (*x*₂) where the subscript 2 stands for component 2 (light gases) in the mixtures. The solubilities of carbon dioxide and ethane in liquid cyclohexanol decrease with temperature, while those of nitrogen increase with temperature. Within the investigated conditions, the solubilities of carbon dioxide and nitrogen increase almost linearly with pressure. The results of cyclohexanol + nitrogen from Lebedeva and Grokholskaya (1971) were also shown in Figure 3 (solid symbols). It appears that the trends of the saturated compositions varying with temperature and pressure agree qualitatively between these two data sources, but several reported values of Lebedeva and Grokholskaya (1971) are not consistent.

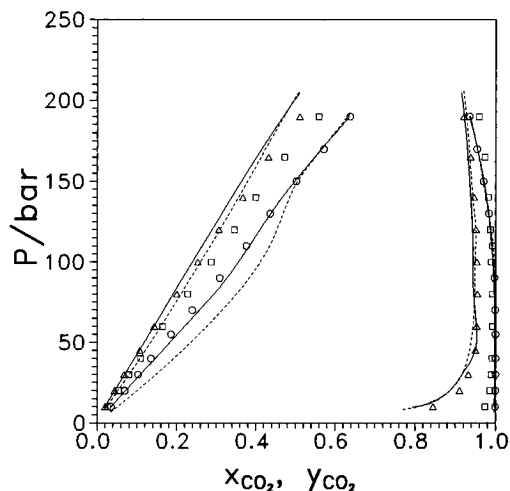
The right portions of Figures 1–3 illustrate the variations of saturated vapor compositions of the light compo-

Table 2. Phase Compositions for Cyclohexanol (1) + Ethane (2)

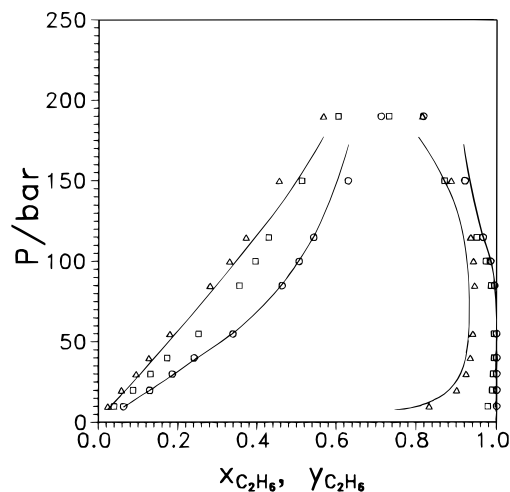
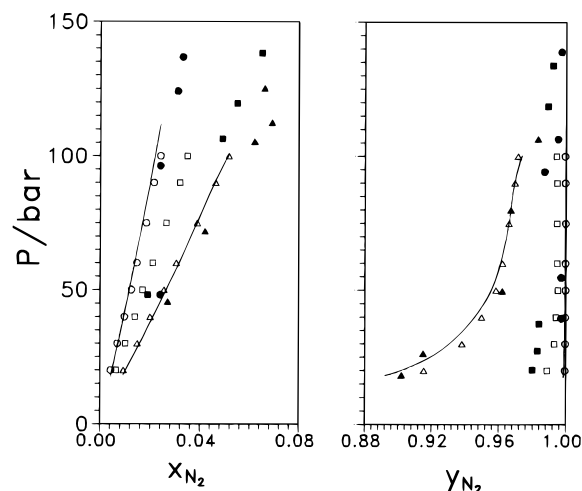
<i>P</i> /bar	<i>y</i> ₁	<i>x</i> ₁	<i>P</i> /bar	<i>y</i> ₁	<i>x</i> ₁
<i>T</i> = 333.15 K					
10.0	0.000177	0.9368	85.0	0.00573	0.537
20.0	0.000170	0.872	100.0	0.0160	0.494
30.0	0.000163	0.814	115.0	0.0347	0.457
40.0	0.000181	0.759	150.0	0.0797	0.370
55.0	0.000340	0.661	190.0	0.182	0.288
<i>T</i> = 393.15 K					
10.0	0.0227	0.9608	85.0	0.0145	0.645
20.0	0.0117	0.9137	100.0	0.0272	0.605
30.0	0.00883	0.869	115.0	0.0505	0.572
40.0	0.00696	0.827	150.0	0.130	0.487
55.0	0.00703	0.748	190.0	0.268	0.396
<i>T</i> = 453.15 K					
10.0	0.168	0.9767	85.0	0.0556	0.719
20.0	0.0996	0.9425	100.0	0.0582	0.670
30.0	0.0761	0.9056	115.0	0.0652	0.629
40.0	0.0659	0.874	150.0	0.114	0.545
55.0	0.0594	0.821	190.0	0.186	0.433

Table 3. Phase Compositions for Cyclohexanol (1) + Nitrogen (2)

<i>P</i> /bar	<i>y</i> ₁	<i>x</i> ₁	<i>P</i> /bar	<i>y</i> ₁	<i>x</i> ₁
<i>T</i> = 333.15 K					
20.0	0.000592	0.99573	60.0	0.000365	0.9853
30.0	0.000508	0.99309	75.0	0.000396	0.9816
40.0	0.000402	0.99032	90.0	0.000605	0.9785
50.0	0.000382	0.9875	100.0	0.000745	0.9759
<i>T</i> = 393.15 K					
20.0	0.0113	0.99370	60.0	0.00540	0.9790
30.0	0.00717	0.9899	75.0	0.00555	0.9736
40.0	0.00596	0.9862	90.0	0.00564	0.9681
50.0	0.00511	0.9831	100.0	0.00601	0.9652
<i>T</i> = 453.15 K					
20.0	0.0847	0.99069	60.0	0.0379	0.9695
30.0	0.0619	0.9852	75.0	0.0342	0.9611
40.0	0.0502	0.9800	90.0	0.0307	0.9537
50.0	0.0417	0.9745	100.0	0.0289	0.9485

**Figure 1.** Pressure–composition diagram for the cyclohexanol (1) + carbon dioxide (2) mixtures: ○, 333.15 K; □, 393.15 K; △, 453.15 K; ---, calculated from the Patel–Teja equation with mixing rule A; - - -, calculated from the Patel–Teja equation with mixing rule B.

nents (*y*₂), indicating that the mole fractions of cyclohexanol (1 - *y*₂) decrease with pressure over the low-pressure regions. A minimum cyclohexanol solubility occurs around (30 to 85) bar at a given equilibrium temperature, except for the 453.15 K isotherm of cyclohexanol + nitrogen. A crossover phenomenon is also exhibited on the saturated vapor isotherms for the systems containing carbon dioxide or ethane. This is not the case for cyclohexanol + nitrogen

**Figure 2.** Pressure–composition diagram for the cyclohexanol (1) + ethane (2) mixtures: ○, 333.15 K; □, 393.15 K; △, 453.15 K; —, calculated from the Patel–Teja equation with mixing rule A.**Figure 3.** Pressure–composition diagram for the cyclohexanol (1) + nitrogen (2) mixtures: (this work) ○, 333.15 K; □, 393.15 K; △, 453.15 K; (Lebedeva and Grokholskaya (1971)) ●, 373.15 K; ■, 423.15 K; ▲, 473.15 K; —, calculated from the Patel–Teja equation with mixing rule A.

because the investigated pressures (up to 100 bar) were well below the critical pressure of these mixtures.

Empirical Correlation

The isothermal saturated compositions of cyclohexanol in the vapor phase (*y*₁) were correlated empirically with the density (ρ) of the pure light components at the equilibrium conditions by

$$\ln y_1 = A + B\rho + C\rho^2 + D\rho^3 \quad (1)$$

where the subscript 1 denotes component 1 (cyclohexanol) in the mixtures. The density of carbon dioxide was calculated from the equation proposed by Huang et al. (1985), and those of ethane and nitrogen were calculated from the Benedict–Webb–Rubin (BWR) equation (Walas, 1985). The calculated results are given in Table 4. This empirical equation could be used to interpolate the saturated vapor compositions within the investigated conditions. Figure 4 shows the results for cyclohexanol + nitrogen.

Henry's Constant

The Krichevsky–Ilinskaya (KI) equation (Krichevsky and Ilinskaya, 1945) is generally applicable to correlating

Table 4. Results of Empirical Correlation for the Saturated Vapor Compositions of Cyclohexanol (y_1)

system ^a	<i>T</i> /K	<i>n</i>	<i>A</i>	<i>B</i>	$10^{-3}C$	$10^{-7}D$	$10^2(\text{AAD})^b$
M1	333.15	12	-7.5550	311.56	-0.73		0.09
	393.15	11	-4.0317	-335.43	53.29		0.35
	453.15	11	-1.6921	-1382.37	393.93	-3.1460	0.66
M2	333.15	10	-8.9422	217.93	30.91		0.19
	393.15	10	-3.6741	-1310.64	403.79	-2.5677	0.91
	453.15	10	-1.5817	-1362.10	390.77	-2.8212	0.61
M3	333.15	8	-6.6550	-1248.60	310.82		0.01
	393.15	8	-3.8673	-1389.76	334.81		0.05
	453.15	8	-1.8953	-1298.96	261.27		0.14

^a M1 = cyclohexanol (1) + carbon dioxide (2), M2 = cyclohexanol (1) + ethane (2), and M3 = cyclohexanol (1) + nitrogen (2). ^b $\text{AAD} = \sum_{k=1}^n |y_{1,k}^{\text{calc}} - y_{1,k}^{\text{exp}}|/n$ where *n* is the number of data points.

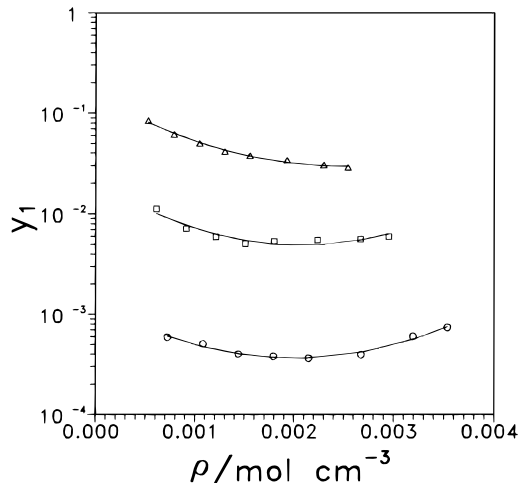


Figure 4. Correlation between the saturated vapor composition of cyclohexanol and the density of nitrogen: ○, 333.15 K; □, 393.15 K; △, 453.15 K; —, calculated from eq 1.

gas solubility data over a wide pressure range. Henry's constant can be obtained by regressing the solubility data to this model:

$$\ln(f_2/x_2) = \ln H_{2,1}^* + [A(x_1^2 - 1)/RT] + \bar{V}_2(P - P_1^{\text{sat}})/RT \quad (2)$$

where $H_{2,1}^*$ is the Henry's constant at the vapor pressure of cyclohexanol (P_1^{sat}), *A* is the Margules constant, and \bar{V}_2 is the partial molar volume of the gas at infinite dilution. In this work, \bar{V}_2 was estimated from the generalized equation developed by Brelvi and O'Connell (1972), and the modified Rackett model (Spencer and Danner, 1972) was employed to calculate the liquid densities. The fugacity of the light component in the vapor phase (f_2) was computed on the basis of the Lewis fugacity rule:

$$f_2 = f_2^0 y_2 \quad (3)$$

where f_2^0 is the fugacity of the pure fluids of component 2 that was calculated from the equation of Huang et al. (1985) for carbon dioxide and the BWR equation (Walas, 1985) for ethane and nitrogen. The values of $H_{2,1}^*$ and the Margules constant were then determined from fitting the KI equation to the solubility data by a least-squares algorithm. Good correlations were obtained as shown in Table 5. Henry's constant increases with temperature for carbon dioxide and ethane, while that of nitrogen decreases with temperature.

Data Reduction with Equations of State

The Peng–Robinson (1976) and Patel–Teja (1982) equations of state with three different types of mixing rules were

Table 5. Parameters of the KI Equation for the Binary Systems of Cyclohexanol (1) + Carbon Dioxide (2), + Ethane (2), or + Nitrogen (2)

component (2)	<i>T</i> /K	$H_{2,1}^*/\text{bar}$	<i>A</i> / (J·mol ⁻¹)	$\bar{V}_2^{\infty}/\text{cm}^3\cdot\text{mol}^{-1}$	AAD ^a / %
carbon dioxide	333.15	316.93	3267.08	44.63	3.4
	393.15	392.95	3394.13	61.89	1.0
	453.15	480.02	4465.81	89.31	2.3
ethane	333.15	175.42	2785.10	58.54	3.5
	393.15	245.86	2885.58	80.75	2.4
	453.15	371.11	4673.48	115.41	4.5
nitrogen	333.15	4442.11	14069.49	43.47	3.0
	393.15	3084.49	11826.94	60.31	1.7
	453.15	2107.14	10757.05	87.14	1.6

$$^a \text{AAD} = (1/n) \sum_{k=1}^n [|(f_2/x_2)_k^{\text{calc}} - (f_2/x_2)_k^{\text{exp}}| / (f_2/x_2)_k^{\text{exp}}]$$

Table 6. Physical Properties and Equation Parameters for the Pure Components

compound	<i>T</i> _c ^a /K	<i>P</i> _c ^a /bar	ω^a	κ^b	ζ_c^c	<i>F</i> ^c
cyclohexanol	625.0	37.5	0.528	1.07277	0.303	1.049936
carbon dioxide	304.1	73.8	0.239	0.72782	0.309	0.707727
ethane	305.4	48.8	0.099	0.52468	0.317	0.561567
nitrogen	126.2	33.9	0.039	0.43438	0.329	0.516798

^a Taken from Reid et al. (1987). ^b κ is a constant in $\alpha(T)$ of the Peng–Robinson equation (Peng and Robinson, 1976) which was correlated with the fluid's acentric factor (ω) for nonpolar fluids: $\kappa = 0.37464 + 1.54226\omega - 0.26992\omega^2$. The values of κ for carbon dioxide, ethane, and nitrogen were estimated from the above generalized correlation, and κ for cyclohexanol was determined in this work. ^c ζ_c is the hypothetical critical compressibility factor as defined by Patel and Teja (1982), and *F* is a constant in $\alpha(T)$ of the Patel–Teja equation (Patel and Teja, 1982). The values of ζ_c and *F* for carbon dioxide, ethane, and nitrogen were taken from Georjoton et al. (1986), and those for cyclohexanol were determined in this work.

applied to correlate the new VLE data. The mixture constants θ_m , including a_m , b_m , and c_m , were calculated from

$$\theta_m = \sum_i \sum_j x_i x_j \theta_{ij} \quad (4)$$

The combining rule of c_{ij} (for the Patel–Teja equation only) was given by

$$c_{ij} = (c_i + c_j)/2 \quad (5)$$

In mixing rule A (one-fluid, one-parameter van der Waals mixing rule), the combining rules of a_{ij} and b_{ij} were defined, respectively, as

$$a_{ij} = (1 - k_{a_{ij}})(a_i a_j)^{0.5} \quad (6)$$

and

$$b_{ij} = (b_i + b_j)/2 \quad (7)$$

where $k_{a_{ij}}$ is a binary interaction constant. When an additional cross-parameter, $k_{b_{ij}}$, was introduced into the combining rule of b_{ij} , i.e.,

$$b_{ij} = (1 - k_{b_{ij}})(b_i + b_j)/2 \quad (8)$$

the mixing rule was designated as mixing rule B (one-fluid, two-parameter van der Waals mixing rule). In mixing rule C, b_{ij} was evaluated from eq 7 and a_{ij} was computed from the composition-dependent combining rule (Panagiotopoulos and Reid, 1986):

$$a_{ij} = [(1 - k_{a_{ij}}) + x_i(k_{a_{ij}} - k_{a_{ij}})](a_i a_j)^{0.5} \quad (9)$$

Table 7. Results of Bubble-Pressure Calculations from the Peng–Robinson and Patel–Teja Equations of State

mixture ^a	mixing rule A			mixing rule B				mixing rule C			
	k_{a12}	100 $\Delta P/P$	100 Δy_1	k_{a12}	k_{b12}	100 $\Delta P/P$	100 Δy_1	k_{a12}	k_{a21}	100 $\Delta P/P$	100 Δy_1
Peng–Robinson Equation of State											
M1	0.1445	15.48	0.88	0.1016	-0.0583	4.63	0.91	0.2230	0.1218	5.87	1.05
M2	0.0944	10.71	2.30	0.0852	-0.0149	7.29	2.80	0.1219	0.0877	7.01	2.35
M3	0.4891	6.97	0.38	0.8633	0.0847	6.32	0.33	0.4995	0.2808	6.92	0.45
grand AAD		11.53	1.23			6.00	1.40			6.55	1.33
Patel–Teja Equation of State											
M1	0.1451	13.95	0.80	0.1246	-0.0496	4.31	0.61	0.2192	0.1181	5.44	0.92
M2	0.0923	5.64	1.10	0.0864	-0.0076	5.10	1.09	0.0921	0.0921	5.64	1.10
M3	0.4287	5.44	0.32	0.4346	0.0029	5.40	0.33	0.4467	0.0980	5.20	0.46
grand AAD		8.80	0.77			4.87	0.70			5.51	0.86

^a M1 = cyclohexanol (1) + carbon dioxide (2), M2 = cyclohexanol (1) + ethane (2), and M3 = cyclohexanol (1) + nitrogen (2).

The optimal cross-parameters (k_{aj} , k_{bj} , and k_{aj}) were determined from the bubble-pressure calculations by minimizing the objective function π with a modified Levenberg–Marquardt algorithm:

$$\pi = \sum_{k=1}^n \left(\frac{|P_k^{\text{calc}} - P_k^{\text{exp}}|}{P_k^{\text{exp}}} + \left| y_{1,k}^{\text{calc}} - y_{1,k}^{\text{exp}} \right| \right) \quad (10)$$

where n is the number of data points, P is the bubble pressure, y is the vapor composition, the subscript k represents the k th point, the superscript calc denotes the calculated value, and the superscript exp denotes the experimental value.

In the phase-equilibrium calculations, the pure-component parameter κ in the Peng–Robinson equation was estimated from the original generalized expression for the light components (carbon dioxide, ethane, and nitrogen), whereas that of cyclohexanol was determined from the vapor pressure data (Perry and Green, 1984). The values of ξ_c and F in the Patel–Teja equation were taken from Georgeton et al. (1986) for the light components, and those of cyclohexanol were obtained by fitting the equation to the vapor pressure data (Perry and Green, 1984). The critical properties and the acentric factor (ω) of the pure compounds listed in the data bank of Reid et al. (1987) were used. Those input variables of the bubble-pressure calculations are given in Table 6. Table 7 shows the results of the bubble-pressure calculations. $\Delta P/P$ and Δy_1 are defined, respectively, as

$$\Delta P/P = \frac{1}{n} \sum_{k=1}^n \frac{|P_k^{\text{calc}} - P_k^{\text{exp}}|}{P_k^{\text{exp}}} \quad (11)$$

$$\Delta y_1 = \sum_{k=1}^n |y_{1,k}^{\text{calc}} - y_{1,k}^{\text{exp}}|/n \quad (12)$$

The tabulated values show that the Patel–Teja equation is superior to the Peng–Robinson equation. As shown in Figures 2 and 3, the simplest mixing rule (mixing rule A) is capable of describing the phase behavior of cyclohexanol + ethane and cyclohexanol + nitrogen except for a few points near the critical point of the ethane-containing mixtures. The additional binary interaction constant used in mixing rule B or C (k_{bj} or k_{aj}) does not make a substantial improvement in the phase-equilibrium calculations for these two systems. Using either mixing rule B or mixing rule C, however, reduces the deviations of the calculated bubble pressures for cyclohexanol + carbon dioxide. Figure 1 indicates that mixing rule A underesti-

mates the bubble pressures of cyclohexanol + carbon dioxide over low to moderate pressures. In general, the Patel–Teja equation with mixing rule B (one-fluid, two-parameter van der Waals mixing rule) yields the best results for the systems studied.

Conclusion

Vapor–liquid equilibrium data were measured for cyclohexanol + carbon dioxide, + ethane, and + nitrogen over a temperature range of (333.15 to 453.15) K and at pressures up to 190 bar. The isothermal solubilities of the gases in cyclohexanol increased with pressure. Within the investigated conditions, the solubilities of carbon dioxide and ethane decrease with temperature, whereas those of nitrogen increase with temperature. The KI equation correlated those gas solubility data well, and Henry's constants of the light gases in cyclohexanol were calculated. While the Patel–Teja equation with the one-fluid, one-parameter van der Waals mixing rule was capable of describing the phase behavior of cyclohexanol + ethane and cyclohexanol + nitrogen, the equation of state with the one-fluid, two-parameter van der Waals mixing rule (mixing rule B) gave the best results of the bubble-pressure calculations for the three systems studied.

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