Solubility of H₂S and CO₂ in N-Octyl-2-pyrrolidone and of H₂S in Methanol and Benzene

Kai Fischer

Laboratory for Thermophysical Properties LTP GmbH, Institute at the University of Oldenburg, FB9-TC, D-26111 Oldenburg, Germany

Jian Chen

Tsinghua University, Dept. of Chemical Engineering, Beijing, 100084 China

Martin Petri

Wacker Chemie GmbH, ITI/LP 27, D-84489 Burghausen, Germany

Jürgen Gmehling

University of Oldenburg, Dept. of Industrial Chemistry, D-26111 Oldenburg, Germany

The solubility of hydrogen sulfide and carbon dioxide in N-octyl-2-pyrrolidone (NOP) was measured up to 1.4 MPa at 303 and 324 K using the static synthetic method. Experimental results were compared to those of the predictive Soave—Redlich—Kwong (PSRK) model, wherein the group interaction parameters for the NMP-group (N-methyl-2-pyrrolidone) were applied to the NOP systems. The reliability of the apparatus used and the applied data treatment were checked by comparing new experimental data for the system hydrogen sulfide + benzene at 304 and 324 K and pressures up to 1.2 MPa, and for the system hydrogen sulfide + methanol at 298 K and up to 0.4 MPa, with published data measured with the analytical method.

Introduction

The knowledge of the solubilities of sour gases like carbon dioxide or hydrogen sulfide in suitable solvents is required for the design of absorption processes for the treatment of natural gas. Physical or chemical solvents can be used. Because of its unique physical properties, such as high polarity, low volatility, miscibility with water and organic solvents, and thermal and chemical stability, *N*-methyl-2-pyrrolidone (NMP) is a physical solvent commonly used for extractive distillation or absorption processes (GAF Corp., 1972). In a previous work (Noll et al., 1996) the binary system water – *N*-methyl-2-pyrrolidone was examined because of its importance as a mixed solvent. The concentration of water in NMP has a significant influence on both the selectivity and the capacity of the solvent mixture (Fischer and Gmehling, 2002; Krummen et al., 2000).

The replacement of the *N*-methyl group by a longer alkyl chain, for example, the ethyl or the octyl group, allows the volatility of the *N*-alkyl-2-pyrrolidone to be reduced, while

keeping the high selectivity and advantageous properties just mentioned. Carbon dioxide and hydrogen sulfide solubilities were measured in N-octyl-2-pyrrolidone (NOP) in this work. The data were used to check whether the PSRK group-contribution equation of state (EOS) (Holderbaum and Gmehling, 1991; Fischer and Gmehling, 1996; Gmehling et al., 1997; Horstmann et al., 2000) can be used to extrapolate the correlation of the existing data for the NMP systems in Lenoir et al. (1971), Murrieta-Guevara and Rodriguez (1984), Murrieta-Guevara et al. (1988), Rivas and Prausnitz (1979a,b) Sweeney (1984), Yarym-Agaev et al. (1980), Byeseda et al., (1985), Rivas (1974) for the H₂S systems, and in (Lenoir et al. (1971), Murrieta-Guevara and Rodriguez (1984), Murrieta-Guevara et al. (1988), Rivas and Prausnitz, (1979a, b), Sweeney (1984), Byeseda et al. (1985), Rivas (1974), Melzer et al. (1989), Wu et al. (1985), Yogish (1991), Usyukin and Shleinikov (1963), Vilcu et al. (1991), Fischer (2002), Zubchenko et al. (1985), Schroedter (1991), Schlichting (1991) for the CO_2 systems to other *N*-alkyl-2-pyrrolidone systems. Test measurements were performed for the solubility of hy-

Correspondence concerning this article should be addressed to J. Gmehling.

drogen sulfide in methanol, and in benzene to validate the indirect static synthetic method and the data treatment procedure by comparing the results to the literature data (Short et al., 1983; Schalk and Onken, private communication, 1973; Bezdel and Teodorovich, 1958; Yorizane et al., 1969; Leu et al., 1992; Preuss and Moerke, 1990) for the $\rm H_2S$ + methanol system, and in Bell (1931), Patyi et al. (1978), Gerrard (1972), Laugier and Richon (1995) for the $\rm H_2S$ + benzene system, which were partly obtained with the direct static analytical method. Except for the $\rm H_2S$ + methanol system, the Henry coefficients derived from the Px data obtained in this work were already published elsewhere together with the values for various other systems (Fischer et al., 1999).

Experimental Studies

Materials

Hydrogen sulfide and carbon dioxide, both with a purity 99.995 mol %, were purchased from Messer Griesheim and used without any further purification. Benzene supplied by Fluka with an initial purity of 99.7 mass %, methanol supplied by Scharlau with an initial purity of 99.99 mass %, and NOP supplied by Hoechst, were distilled and degassed after drying over a molecular sieve by vacuum distillation, as described before (Fischer and Gmehling, 1994).

Apparatus and procedure

The apparatus used was described in detail by Fischer and Gmehling (1994). The modifications of the experimental setup as well as the raw data treatment, which is required for the indirect experimental method for determining the gas solubility in the liquid phase, are outlined by Fischer and Wilken (2001). The applied indirect experimental method provides

Table 1. Experimental Isothermal Px Data for the Hydrogen Sulfide (1)-Methanol or Benzene (2) System

Methanol 298.15 K		Benzene					
		304.30	K	323.50 K			
$P(\times 10^5 \text{ Pa})$	<i>x</i> ₁	$P(\times 10^5 \text{ Pa})$	<i>x</i> ₁	$P(\times 10^5 \text{ Pa})$	x_1		
0.183	0.0000	1.024	0.0413	1.322	0.0339		
0.230	0.0028	1.161	0.0479	2.133	0.0629		
0.375	0.0078	1.696	0.0741	3.173	0.1001		
0.610	0.0153	2.544	0.1159	4.271	0.1393		
0.695	0.0152	3.550	0.1657	5.642	0.1881		
1.035	0.0261	4.299	0.2029	6.730	0.2262		
1.061	0.0260	5.914	0.2826	7.797	0.2632		
1.461	0.0378	7.400	0.3548	8.924	0.3018		
1.478	0.0378	8.740	0.4183	10.091	0.3409		
1.903	0.0502	9.443	0.4508	11.006	0.3711		
1.908	0.0502	10.260	0.4879	11.805	0.3969		
2.320	0.0643						
2.328	0.0643						
2.749	0.0800						
2.766	0.0800						
3.165	0.0930						
3.173	0.0930						
3.194	0.0930						
3.249	0.0930						
3.635	0.1060						
3.652	0.1060						
4.140	0.1231						

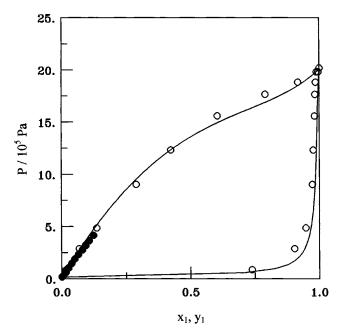


Figure 1. Experimental isothermal Px(y) data for the hydrogen sulfide (1) – methanol (2) system at 298 K.

From Leu et al. (1992) (○) and from this work (●) compared to predictions using the PSRK method.

the system pressure P of a mixture with known composition charged into a thermostated equilibrium cell at temperature T. The global feed composition is known precisely, while the

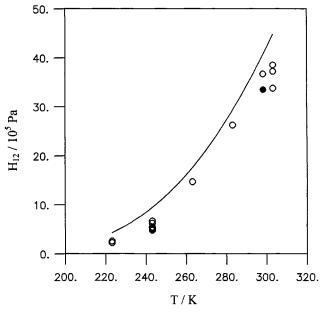


Figure 2. Experimental Henry coefficients for hydrogen sulfide (1) in methanol (2).

From Short et al. (1983); Schalk and Onken, private communication (1973); Bezdel and Teodorovich (1958) (\bigcirc) ; and derived from the isothermal Px data from this work (\bullet) compared to predictions using the PSRK method.

Table 2. Pure Component Parameters for EOS-Calculations

Compound	Critical Data		Mathias-Copeman Constants			VdW Properties	
	T_c (K)	P_c (MPa)	c_1	c_2	c_3	R	Q
Carbon dioxide	304.2	7.376	0.82524	0.25155	-1.70390	1.3000	1.120
Hydrogen sulfide	373.2	8.937	0.63564	-0.45039	1.68370	1.2350	1.202
Benzene	562.1	4.894	0.83560	-0.37500	0.97150	3.1878	2.400
Methanol	512.6	8.096	1.43710	-0.79940	0.32780	1.4311	1.432
NOP	748.2	2.371	1.19778	0.00000	0.00000	8.7018	6.980
NMP	721.7	4.519	1.02050	0.00000	0.00000	3.9810	3.200

Table 3. Binary Interaction Parameters Fitted to the Isothermal Experimental Data

	T (K)	SRK + PSRK UNIC	SRK + Classic Mixing Rules	
		$A_{12} (kJ \cdot mol^{-1})$	$A_{21} (kJ \cdot mol^{-1})$	$\overline{k_{12}}$
Hydrogen sulfide (1) + methanol (2)	298.15	27.590	-1.6170	0.039466
Hydrogen sulfide (1) + benzene (2)	304.30	-2.0858	3.1927	0.003946
Hydrogen sulfide (1) + benzene (2)	323.50	-2.3597	3.5870	-0.003416
Hydrogen sulfide $(1) + NOP(2)$	306.70	-2.5107	2.5107	-0.146710
Hydrogen sulfide $(1) + NOP(2)$	323.60	-2.4056	2.4055	-0.138160
Carbon dioxide $(1) + NOP(2)$	303.40	15.837	-2.4534	0.010196
Carbon dioxide $(1) + NOP(2)$	323.60	7.2570	-2.5357	0.006809

composition of the liquid phase is obtained by subtracting the molar amount of all compounds in the vapor phase from the total molar amount loaded in the equilibrium cell. Further details about the data treatment method are described by Fischer and Wilken (2001). The uncertainty the raw data treatment introduced depends mainly on the relative molar amount of the gaseous compound in the vapor phase. Therefore the equilibrium cell was charged almost completely full with the solvent, leaving a relatively small vapor space. The overall uncertainty of the liquid-phase composition was estimated to be $x_1 = \pm 0.0001$, taking into account the accuracy of the feed composition and the accuracy limited by the raw data treatment method. The DIPPR density correlation (Daubert et al., 1998), or experimental values in the case of NOP (914.7 kg/m³ at 303.15 K, and 899.7 kg/m³ at 323.15 K), were used to calculate the amounts of the liquid solvent and of the gas loaded in the equilibrium cell. The temperature was measured with an uncertainty of ± 0.02 K, and the pressure with an uncertainty of ± 0.05 %, or ± 0.2 kPa.

Experimental results and discussion

The results for the solubility of hydrogen sulfide in methanol at 298 K and in benzene at 304 K and at 324 K are listed in Table 1. Since experimental data for the solubility of hydrogen sulfide in methanol at 298 K are available (Leu et al., 1992), they were used for comparison (Figure 1). The solid line represents the calculation using PSRK. The experimental data agree quite well with each other and with the PSRK prediction. The Px data from this work were also correlated with the SRK EOS, where classical mixing rules and also the PSRK mixing rule combined with the UNIQUAC model were used. The pure-component parameters used for the EOS calculations data are listed in Table 2. The fitted binary interaction parameters are given in Table 3. The PSRK group interaction parameters are published elsewhere (Holderbaum and Gmehling, 1991; Fischer and Gmehling, 1996; Gmehling et al., 1997; Horstmann et al., 2000).

Table 4. Henry Coefficients for Solubility of Hydrogen Sulfide or Carbon Dioxide (1) in NOP or Methanol or Benzene (2)*

H_{12} (MPa)
3.35
1.96
2.69
0.70
1.15
5.00
6.67

^{*}Derived from Experimental Isothermal Px Data by Correlation with the SRK EOS and the UNIQUAC G^E Model with the PSRK Mixing Rule.

The fugacity coefficient of H₂S at infinite dilution and the Henry coefficient were calculated from this zero partial pressure correlation of H₂S. This value is given in Table 4, along with the Henry coefficients for all the other systems measured, and plotted in Figure 2 together with several literature data, and with the predicted temperature dependence of the Henry coefficient by PSRK. Again, all the data agree quite well with each other. The predicted Henry coefficients are slightly larger than the experimental ones. Also VLE data for the H_2S + benzene system are available in the literature (Laugier and Richon, 1995), and are compared with our values at ca. 323 K in Figure 3. The data agree very well with each other and with the predicted results of the PSRK model. The results from the literature (Leu et al., 1992; Laugier and Richon, 1995) shown in Figures 1 and 3 were obtained with the direct static analytical method. As for the previous system, the Henry coefficients from the literature and from our data correlation are plotted vs. the temperature and compared with the results of the PSRK model in Figure 4. Except for one value at 298 K (Patyi et al., 1978), the experimental data are in good agreement. The Henry coefficients predicted with PSRK are lower than the experimental values, but its temperature dependence is correct. From the preced-

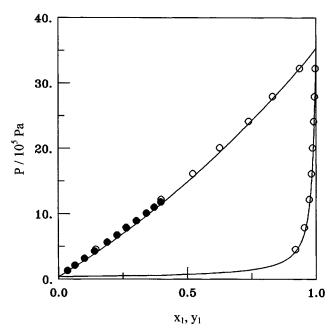


Figure 3. Experimental isothermal Px(y) data for the hydrogen sulfide (1) – benzene (2) system at 323 K.

From Laugier and Richon (1995) (○) and from this work (●) compared to predictions using the PSRK method.

ing results and comparison, it can be concluded that the indirect static synthetic method and the data treatment procedure used in this work provide reliable gas solubility data.

In the next step, carbon dioxide and hydrogen sulfide solubilities were measured in NOP. The experimental data are given in Table 5. For each temperature, the experimental *Px* data are plotted in Figures 5 and 6 (H₂S solubilities) and Figures 7 and 8 (CO₂ solubilities). At the same time, the results of three different approaches are shown together with the experimental data: (1) solid line: correlation with the PSRK/UNIQUAC model; (2) dashed line: correlation with

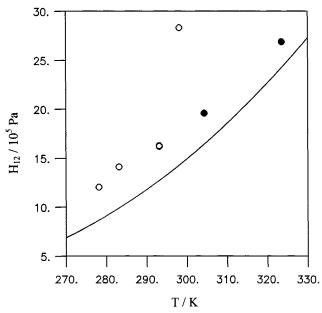


Figure 4. Experimental Henry coefficients for hydrogen sulfide (1) in benzene (2).

From Bell (1931); Patyi et al. (1978); Gerrard (1972) (\bigcirc) and derived from the isothermal Px data from this work (\bullet) compared to predictions using the PSRK method.

the SRK EOS and classic mixing rules; (3) dotted line: prediction with the PSRK/UNIFAC model. The fitted binary interaction parameters for correlation approaches (1) and (2) are given in Table 3. In order to apply the PSRK group contribution EOS to the NOP systems, NOP was subdivided into the following groups: the NMP group and seven ${\rm CH_2}$ groups. This allowed us to apply the already available NMP parameters for systems with NOP. The correlation with the PSRK/UNIQUAC model allows to describe all the data within experimental uncertainly. But the SRK EOS with the classic mixing rules is not flexible enough to correlate the data for the ${\rm H_2S+NOP}$. The calculated bubble-point pressures are lower than the experimental values. The opposite

Table 5. Experimental Isothermal Px data for the Hydrogen Sulfide or Carbon Dioxide (1) - N-Octyl-2-pyrrolidone (2) System

Hydrogen Sulfide				Carbon Dioxide			
306.70 K		323.60 K		303.40 K		323.60 K	
P (×10 ⁵ Pa) 1.062	0.1365	P (×10 ⁵ Pa) 1.271	0.1050	P (×10 ⁵ Pa) 1.068	0.0217	P (×10 ⁵ Pa) 1.139	0.0170
1.529 1.990	0.1870 0.2323	1.712 2.046	0.1376 0.1611	1.531 2.176	0.0310 0.0437	2.433 3.816	0.0360 0.0560
2.515 3.268	0.2792 0.3392	2.515 3.024	0.1925 0.2247	2.962 3.892	0.0589 0.0767	5.266 6.471	0.0765 0.0934
4.032	0.3925	3.541	0.2554	5.149	0.1001	7.537	0.1081
5.155 5.947	0.4604 0.5021	4.082 4.778	0.2858 0.3223	6.358 7.900	0.1221 0.1493	9.122 10.829	0.1296 0.1524
6.752 7.952	0.5403 0.5907	5.572 6.277	0.3609 0.3926	9.179 10.342	0.1712 0.1906	12.414 13.803	0.1729 0.1908
9.006 9.844	0.6298 0.6579	7.082 8.002	0.4263 0.4618	11.374 12.472	0.2075 0.2251		
10.506 11.099	0.6787 0.6963	8.716 9.663 10.444	0.4874 0.5191 0.5434	13.223	0.2369		

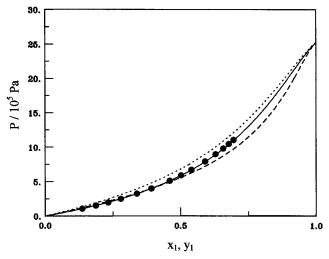


Figure 5. Experimental (• this work) and calculated (

PSRK/UNIQUAC; --- SRK/quadratic mixing rule; ··· PSRK/UNIFAC) VLE behavior for the hydrogen sulfide (1) + NOP (2) system at 307 K.

trend is observed when comparing the experimental data with the predicted results of the PSRK/UNIFAC model, for which the bubble-point line is slightly moved toward higher pressures. The different calculations for the $\rm CO_2 + \rm NOP$ system show smaller differences, except in the critical region. Since no experimental data are available at pressures around the critical pressure of $\rm CO_2$, there is no hint to which kind of calculated critical behavior meets the truth. Both correlations and the prediction allow the experimental solubility data of $\rm CO_2$ to be represented very well.

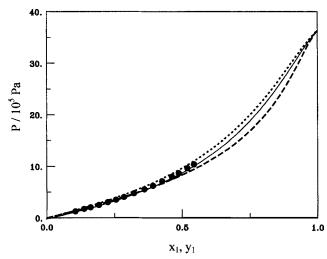


Figure 6. Experimental (● this work) and calculated (

PSRK/UNIQUAC; --- SRK/quadratic mixing rule; ···· PSRK/UNIFAC) VLE behavior for the hydrogen sulfide (1) + NOP (2) system at 324 K.

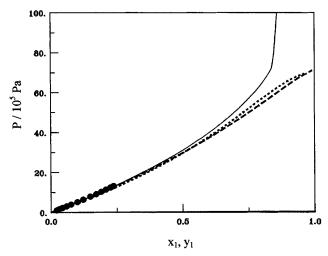


Figure 7. Experimental (• this work,) and calculated (
PSRK/UNIQUAC; --- SRK/quadratic mixing rule; ···· PSRK/UNIFAC) VLE behavior for the carbon dioxide (1) + NOP (2) system at 303 K.

The Henry coefficients for H_2S in NOP are plotted vs. the temperature in Figure 9. The solid line represents the PSRK prediction for this system. The available data from the literature for H_2S solubilities in NMP are plotted in the same diagram along with the dotted line for the PSRK prediction for the H_2S+NMP system. All the results are quite similar for this system. This is not the case for the $CO_2+(NOP\ or\ NMP)$ system, as can be seen in Figure 10. In this case, both the predicted line for the PSRK model and the experimental data differ for the two solvents NMP and NOP. With an increasing number of CH_2 groups in the N-alkyl chain, the solubility

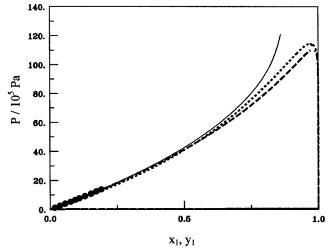


Figure 8. Experimental (● this work) and calculated (——PSRK/UNIQUAC; --- SRK/quadratic mixing rule; ···· PSRK/UNIFAC) VLE behavior for the carbon dioxide (1) + NOP (2) system at 324 K.

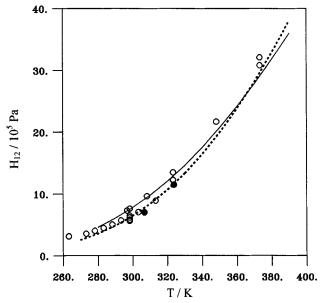


Figure 9. Experimental (● this work) and predicted (______ PSRK/UNIFAC) Henry coefficients for hydrogen sulfide (1) in NOP (2).

Experimental (Lenoir et al., 1971; Murrieta-Guevara and Rodriguez, 1984; Murrieta-Guevara et al., 1988; Rivas and Prausnitz, 1979a,b; Sweeney, 1984; Yarym-Agaev et al., 1980; Byeseda et al., 1985; Rivas, 1974) (O) and predicted (···· PSRK/UNIFAC) Henry coefficients for hydrogen sulfide (1) in NMP (2) as a function of temperature.

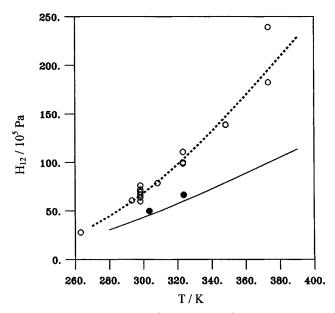


Figure 10. Experimental (• this work) and predicted (____ PSRK/UNIFAC) Henry coefficients for carbon dioxide (1) in NOP (2).

Experimental (Lenoir et al., 1971; Murrieta-Guevara and Rodriguez, 1984; Murrieta-Guevara et al., 1988; Rivas and Prausnitz, 1979a,b; Sweeney, 1984; Byeseda et al., 1985; Rivas, 1974; Melzer et al., 1989; Wu et al., 1985; Yogish, 1991; Usyukin and Shleinikov, 1963; Vilcu et al., 1991; Zubchenko et al., 1985) (O) and predicted (···· PSRK/UNIFAC) Henry coefficients for carbon dioxide (1) in NMP (2) as a function of temperature.

of CO_2 in N-alkyl-2-pyrrolidone increases significantly. This is not the case for $\mathrm{H}_2\mathrm{S}$, and thus the chain length of the N-alkyl group has a strong impact on the selectivity difference between the two sour gases. The PSRK method is a reliable estimation method for all the systems investigated in this article.

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