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Solubility of Carbon Dioxide in Aqueous Mixtures of *N*-Methyldiethanolamine + Piperazine + Sulfolane

Masih Hosseini Jenab,[†] Majid Abedinzadegan Abdi,* Seyed Hesam Najibi,[‡] Mehdi Vahidi,[†] and Naser Seyed Matin[†]

Research Institute of Petroleum Industry (R.I.P.I.), National Iranian Oil Company (N.I.O.C.),
P.O. Box 18745-4163, Tehran, Iran, and Petroleum University of Technology, Research & Development
Department of NIGC

The solubilities of carbon dioxide are reported in mixed solvents containing (1.68 to 3.00) kmol/m^3 (*N*-methyldiethanolamine), (0.36 to 1.36) kmol/m^3 (piperazine), (0.36 to 1.36) kmol/m^3 (tetramethylensulfone or sulfolane), and water for temperatures from (40 to 70) °C and CO_2 partial pressures in the range of (30 to 3900) kPa. The results show that piperazine has an effect on the CO_2 solubility and sulfolane behaves as a physical solvent.

Introduction

Aqueous alkanolamine solutions are widely used for the removal of acid gases such as CO_2 and H_2S from gas streams. Examples of such streams include natural gases, synthesis gases from the gasification of coal and heavy oils, and tail gases from sulfur plants and petroleum chemical plants. Frazier and Kohl[1] first described the use of *N*-methyldiethanolamine (MDEA) solutions. MDEA has a stable structure and does not degrade readily, so it does not cause corrosion problems for carbon steel. Because of chemical reactions in the liquid phase and the strong deviation from ideality, the thermodynamic description of aqueous systems containing alkanolamines such as MDEA and sour gases such as CO_2 is a difficult task. Reliable experimental data for the solubility of acid gases in aqueous solubility containing alkanolamines are required to develop and test thermodynamic models used to describe vapor−liquid equilibria in these systems. However, reliable data on these systems are often scarce or scattered. Thus, the solubility of CO_2 in aqueous solutions containing MDEA, tetramethylensulfone (TMS) or sulfolane, and piperazine (PZ) for a wide range of concentrations and temperatures was measured. In recent years, CO_2 equilibrium solubility in MDEA-based solutions has been reported. Jou et al.[2] reported the solubility of H_2S and CO_2 in (1.0, 2.0, and 4.28) kmol/m^3 aqueous MDEA solutions at temperatures and acid gas partial pressures ranging from (40 to 120) °C and (0.001 to 6600) kPa, respectively. Chakaravarty[3] presented the solubility data of H_2S and CO_2 in aqueous mixtures of MDEA with monoethanolamine (MEA) or diethanolamine (DEA). Chakma et al.[4] determined the solubility of CO_2 in (1.69 and 4.28) kmol/m^3 aqueous MDEA solutions over a range of temperatures from (100 to 200) °C and partial pressures from (172 to 4929) kPa. Austgen et al.[5] reported the CO_2 solubility in 2.0 kmol/m^3 MDEA plus 2.0 kmol/m^3 MEA or a DEA aqueous solution at (40 and 80)°C for CO_2 partial pressures from (0.05 to 315) kPa. MacGregor et al.[6] determined the CO_2 solubility data in 20.9 mass % MDEA, 30.5 mass % TMS, and 48.6 mass % water. Shen and Li[7]- and Li and Shen[8] measured the CO_2 solubility in an aqueous mixture of MDEA and MEA at a total amine concentration of 30 mass %. Xu et al.[9] measured the solubility data for CO_2 in 4.28 kmol/m^3 MDEA with the PZ concentration ranging from (0 to 0.515) kmol/m^3 and the CO_2 partial pressure ranging from (3.83 to 76.77) kPa. Liu et al.[10] determined the CO_2 solubility in an aqueous mixture of MDEA and PZ with the CO_2 partial pressure ranging from (13.16 to 935.3) kPa. Bishnoi and Rochelle[11] showed that PZ has a large effect on solubility when the ratio of total carbon dioxide to PZ is less than 1.

PZ is an effective component for industrial CO_2 removal processes; however, very limited equilibrium data on the solubility of CO_2 in aqueous blends of MDEA with PZ were found in the literature. TMS, however, decreases the solubility of CO_2 in MDEA solutions but has a strong effect on the solubility of sulfur compounds, especially COS and CS_2 in MDEA solutions as shown by Nasir.[12]

This paper presents measurements of the solubility of CO_2 in 1.68 kmol/m^3 MDEA + 0.84 kmol/m^3 PZ + 0.84 kmol/m^3 TMS, 2 kmol/m^3 MDEA + 0.68 kmol/m^3 PZ + 0.68 kmol/m^3 TMS, and 2.5 kmol/m^3 MDEA + 0.43 kmol/m^3 PZ + 0.43 kmol/m^3 TMS at temperatures ranging from (40 to 70) °C and CO_2 partial pressures ranging from (30 to 3900) kPa.

Experimental Apparatus and Procedure

The solubility measurements presented here were carried out in a modified autoclave reactor shown schematically in Figure 1. The reactor consists of an electrically heated 1-L stainless steel cylindrical tank with a magnetically coupled stirrer on the top (model 4842, Parr Instruments Co., Moline, IL). There are valves for the inlet of gas and liquid and a connection to a vacuum pump. A calibrated thermocouple (PT100, Jumo Process Control Inc., Fulda, Germany) inserted into the cell measured the temperature to an uncertainly of ±0.1 K. A water bath was also used to adjust the temperature of the cell more accurately. A calibrated pressure transmitter (model PTX 1400, Druck Messtechnik GmbH, Germany) measured the

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* To whom correspondence should be addressed. E-mail: abdi@engr.mun.ca. Present address: Faculty of Engineering and Applied Science, Memorial University of Newfoundland, St. John's, NL, Canada A1B 3X5.

[†] National Iranian Oil Company.

[‡] Petroleum University of Technology.

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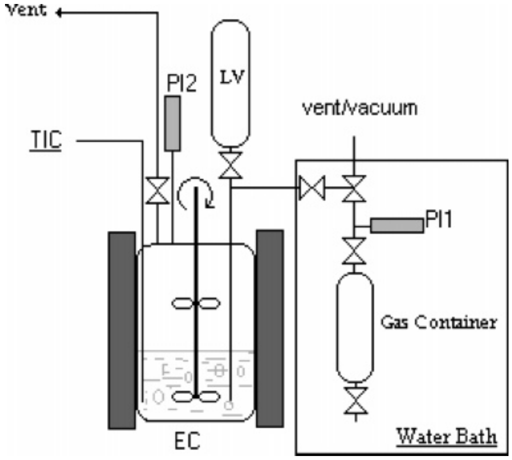

Figure 1. Schematic diagram of experimental equipment: EC, equilibrium cell; LV, liquid vessel; PI1, pressure indicator no. 1; PI2, pressure indicator no. 2; TIC, thermal indicator and controller.

Table 1. Comparison of Solubility Data of CO_2 in Various Amine Solutions at 25, 40, 50, and 70 °C between This Work and Literature Data

t/°C	P_{CO_2}/kPa	$\alpha_{CO_2(\text{this work})}$	$\alpha_{CO_2(\text{lit})}$	dev/%[d]
2.0 kmol/m^3 MDEA				
25.0	181	1.04	1.025[a]	1.46
	698	1.10	1.146[a]	−4.08
	2040	1.34	1.308[a]	2.45
				AAD = 2.66
40.0	101	0.86	0.866[a]	−0.69
	294	1.00	0.990[a]	1.01
	640	1.06	1.083[a]	−2.12
				AAD = 1.27
70.0	447	0.85	0.841(a)	1.07
	993	1.03	1.011(a)	1.88
	2320	1.11	1.147[a]	−3.23
				AAD = 2.06
2.5 kmol/m^3 MDEA				
40.0	138	0.91	0.911[b]	−0.11
3.75 kmol/m^3 MDEA + 1.55 kmol/m^3 PZ				
50.0	90	0.51	0.525[c]	−2.85
4.77 kmol/m^3 MDEA + 0.53 kmol/m^3 PZ				
50.0	90	0.40	0.415[c]	−3.61
1.35 kmol/m^3 MDEA + 0.35 kmol/m^3 PZ				
50.0	502	0.96	0.936[c]	2.56
	579	0.99	0.955[c]	3.66
70.0	240	0.77	0.759[c]	1.45
				AAD = 2.56
1.53 kmol/m^3 MDEA + 0.17 kmol/m^3 PZ				
50.0	276	0.89	0.851[c]	4.58
	661	1.01	0.980[c]	3.06
70.0	147	0.61	0.641[c]	−4.84
	413	0.79	0.811[c]	−2.59
	680	0.85	0.876[c]	−2.97
				AAD = 3.61

[a] Jou et al.[2] [b] Shen et al.[7] [c] Liu et al.[10] [d] dev = $(\alpha_{CO_2(\text{lit})} - \alpha_{CO_2(\text{this work})})/\alpha_{CO_2(\text{lit})} \times 100$.

Table 2. Solubility of CO_2 in Various Mixtures of MDEA + PZ Solutions

	3.00 kmol/m^{3}MDEA + 0.36 kmol/m^3 PZ		2.50 kmol/m^3 MDEA + 0.86 kmol/m^3 PZ		2.00 kmol/m^3 MDEA + 1.36 kmol/m^3 PZ	
t/°C	α_{CO_2}	P_{CO_2}/kPa	α_{CO_2}	P_{CO_2}/kPa	α_{CO_2}	P_{CO_2}/kPa
40	0.4269	33.99	0.4268	27.79	0.5611	30.54
	0.6398	73.98	0.8063	135.34	0.8261	120.17
	0.8365	203.60	0.9426	344.94	0.9588	284.96
	0.9148	384.24	0.9903	555.23	1.0131	553.85
	0.9686	688.98	1.0234	788.27	1.0547	783.44
	0.9872	871.69	1.0544	1059.23	1.0823	1152.99
	0.9991	1030.27	1.0842	1433.60	1.1516	1703.87
	1.0364	1403.96	1.1184	1825.91	1.195	2063.09
	1.0626	1810.74	1.1607	2266.48	1.2285	2495.38
	1.0935	2225.80	1.2171	2810.47	1.2561	2688.43
	1.1607	2869.07	1.2606	3258.62	1.2976	3168.99
	1.2067	3268.27	1.3147	3938.43	1.3613	3673.68
	1.2817	3850.87				
55	0.2268	42.95	0.3221	38.82	0.4597	45.71
	0.4339	95.35	0.5290	106.38	0.613	115.35
	0.4377	102.94	0.6854	236.00	0.7235	233.25
	0.6269	220.84	0.7846	427.68	0.8481	535.23
	0.7211	357.35	0.8516	682.78	0.8872	750.34
	0.8057	601.42	0.8821	910.99	0.9273	1017.86
	0.8522	845.49	0.9266	1281.92	0.9613	1306.74
	0.8979	1238.49	0.9746	1782.47	0.9850	1736.28
	0.9405	1641.82	0.9959	2108.59	1.0064	2092.04
	0.9727	1967.25	1.0258	2465.73	1.0394	2548.64
	1.0142	2452.63	1.0694	2990.42	1.0675	2987.66
	1.0552	2943.53				
70	0.2823	72.95	0.2817	41.23	0.3811	51.57
	0.4286	157.75	0.4500	99.83	0.5603	144.65
	0.5893	326.67	0.5896	214.29	0.7202	336.32
	0.6889	514.89	0.6968	385.27	0.7929	589.35
	0.7603	735.52	0.7769	616.24	0.8354	832.05
	0.8085	987.18	0.8284	869.97	0.9025	1311.22
	0.8508	1245.73	0.8847	1276.75	0.9359	1786.96
	0.8909	1644.24	0.9073	1640.10	0.9603	2149.61
	0.9249	2014.48	0.9313	2014.48	0.9817	2521.24
	0.9462	2476.47	0.9545	2408.85	1.0250	2996.97
	0.9966	2922.50	1.0142	3023.17		

pressure of the cylinder and gas container with an uncertainty of ±0.70 kPa. The compositions of the liquid were analyzed by a gas chromatograph (model 3800, Varian-Chrompack, Varian Inc., CA) using a flame ionization detector (FID) and a 10-m × 0.5-mm DB-WAX capillary column.

Initially, a vacuum was applied to the reactor effectively to generate absolute pressures below 13.3 Pa. Then, the reactor was charged with 200 mL of liquid. The temperature was then adjusted to the desired value through the external heating jackets. In this state, the liquid exists under its own vapor pressure, P_v. The procedure adopted by Park and Sandall[13] was followed, and a known quantity of CO_2, n_{CO_2}, was introduced into the reactor from the gas container of known volume:

$$n_{CO_2} = \frac{V_T}{RT_a}\left(\frac{P_1}{Z_1} - \frac{P_2}{Z_2}\right) \qquad (1)$$

where V_T denotes the volume of the gas container, Z_1 and Z_2 are the compressibility factors corresponding to the initial pressure P_1 and the final pressure P_2 in the gas container before and after transferring the CO_2, and T_a is the ambient temperature. Compressibility factors were calculated using several equations of state including the modified Benedict−Webb−Robin (MBWR), Soave−Redlich−Kwong (SRK), Peng−Robinson (PR), and a number of others available within the HYSYS process simulator. All equations of state produced very similar results with a maximum inconsistency of ±1.2%. The calculated Z values for the range of pressure and temperature conditions tested in this paper were compared with those from the reported density data by Starling,[14] and the error was found to be less than ±0.5%. PR was used for all compressibility coefficient calculations. After transferring the CO_2 to the reactor, the stirrer was turned on, and equilibrium was attained in about 30 min. Then, the equilibrium pressure P_{CO_2} was calculated as

$$P_{CO_2} = P_T - P_V \qquad (2)$$

where P_T denotes the total pressure. The moles of remain-

Table 3. Solubility of CO_2 in Various Mixtures of MDEA + TMS

t/°C	3.00 kmol/m^{3}MDEA + 0.36 kmol/m^3 TMS		2.50 kmol/m^3 MDEA + 0.86 kmol/m^3 TMS		2.00 kmol/m^3 MDEA + 1.36 kmol/m^3 TMS	
	α_{CO_2}	P_{CO_2}/kPa	α_{CO_2}	P_{CO_2}/kPa	α_{CO_2}	P_{CO_2}/kPa
40	0.3328	38.82	0.2902	38.20	0.2242	38.82
	0.7384	208.43	0.3873	52.61	0.4273	133.96
	0.8340	422.16	0.6042	148.44	0.564	373.9
	0.8785	679.33	0.6359	179.54	0.6855	735.87
	0.9015	908.23	0.7462	500.14	0.7541	1086.8
	0.9199	1166.78	0.7498	524.89	0.7844	1356.38
	0.9718	1580.46	0.7766	776.61	0.7988	1622.52
	1.0079	1976.90	0.7907	963.39	0.8679	2200.29
	1.0467	2396.79	0.7921	1023.44	0.9059	2539.51
	1.0887	2769.79	0.8042	1196.5	0.9493	2881.48
	1.1029	2890.44	0.8062	1229.52	0.9827	3120.73
			0.8097	1298.54		
			0.8741	1719.73		
			0.9238	2155.47		
			0.9671	2535.37		
			1.0156	2900.79		
55	0.1650	58.12	0.1021	38.82	0.0597	45.71
	0.3087	102.25	0.2590	106.38	0.213	115.35
	0.4646	184.98	0.4654	236.00	0.4035	233.25
	0.5855	330.46	0.6046	427.68	0.5781	535.23
	0.6658	539.37	0.6716	682.78	0.6572	750.34
	0.7401	790.33	0.7221	910.99	0.7173	1017.86
	0.7881	1104.73	0.7766	1281.92	0.7613	1306.74
	0.8438	1417.06	0.8446	1782.47	0.795	1736.28
	0.8983	1841.77	0.8759	2108.59	0.8264	2092.04
	0.9591	2299.57	0.9158	2465.73	0.8694	2548.64
	0.9809	2724.97	0.9594	2990.42	0.8875	2987.66
	1.0122	3151.06				
70	0.1583	66.74	0.2381	99.83	0.2501	128.10
	0.1690	70.88	0.3683	193.60	0.3435	227.39
	0.4087	214.29	0.4788	337.01	0.4019	336.32
	0.5161	355.63	0.5566	534.40	0.4519	475.59
	0.6067	567.98	0.6248	701.05	0.5136	698.96
	0.6854	807.92	0.6690	894.79	0.5578	968.56
	0.7752	1222.28	0.7283	1249.17	0.6423	1517.37
	0.8072	1590.46	0.7540	1467.00	0.7031	1865.55
	0.8447	1993.11	0.8199	1811.09	0.7627	2236.49
	0.8716	2325.43	0.8723	2239.24	0.8193	2654.99
	0.9038	2717.73	0.9300	2698.43	0.8701	3044.54
			0.9316	2981.80		

Table 4. Solubility of CO_2 in Various Mixtures of MDEA + PZ + TMS Solutions

t/°C	1.68 kmol/m^{3}MDEA + 0.84 kmol/m^3 PZ + 0.84 kmol/m^3 TMS		2.00 kmol/m^3 MDEA + 0.68 kmol/m^3 PZ + 0.68 kmol/m^3 TMS		2.50 kmol/m^3 MDEA + 0.43 kmol/m^3 PZ + 0.43 kmol/m^3 TMS	
	α_{CO_2}	P_{CO_2}/kPa	α_{CO_2}	P_{CO_2}/kPa	α_{CO_2}	P_{CO_2}/kPa
40	0.3844	40.22	0.5266	50.44	0.4763	45.71
	0.4422	67.08	0.7046	177.40	0.6901	125.69
	0.5108	251.17	0.7584	329.08	0.8043	285.65
	0.5541	493.17	0.8064	654.51	0.8754	602.80
	0.5790	762.07	0.8244	908.23	0.9130	984.07
	0.5967	1028.89	0.8422	1139.89	0.9255	1136.45
	0.6118	1252.96	0.8524	1288.89	0.9844	1620.45
	0.6637	1752.14	0.9065	1686.64	1.0353	2134.10
	0.7083	2136.86	0.9506	2091.35	1.0780	2538.13
	0.7531	2524.34	0.9993	2509.86	1.1091	2800.81
	0.7928	2836.67	1.0463	2875.28		
55	0.2646	36.06	0.3120	40.89	0.3463	50.54
	0.3902	100.87	0.5455	141.55	0.5361	118.11
	0.4544	241.52	0.6424	316.67	0.6788	257.38
	0.4990	415.26	0.6948	546.95	0.7720	484.21
	0.5320	695.19	0.7327	888.93	0.8277	776.54
	0.5562	950.29	0.7550	1184.02	0.8647	1090.94
	0.5776	1246.07	0.7664	1418.44	0.8825	1275.03
	0.6082	1649.41	0.8112	1792.13	0.9432	1662.51
	0.6637	2056.19	0.8491	2169.26	0.9804	2003.10
	0.7044	2399.54	0.8919	2605.01	1.0294	2441.60
	0.7351	2751.17	0.9346	3018.68	1.0853	2965.60
	0.7824	3154.51				
70	0.2040	82.60	0.0996	30.20	0.2323	53.64
	0.3211	190.15	0.3611	130.17	0.3831	135.00
	0.4321	414.92	0.4870	292.88	0.4945	263.93
	0.4737	665.20	0.5340	559.71	0.5865	425.95
	0.5158	953.39	0.6112	802.40	0.6567	636.93
	0.5444	1356.04	0.6645	1225.04	0.7018	863.76
	0.5865	1746.97	0.6881	1527.03	0.7767	1404.30
	0.6357	2141.34	0.7616	2010.34	0.8420	1985.52
	0.6850	2558.47	0.8025	2439.19	0.8722	2350.94
	0.7323	2948.70	0.8495	2858.38	0.9003	2721.87
			0.8747	3145.20	0.9319	3105.90

ing CO_2 in the gas phase $n^{g}_{CO_2}$ was determined from

$$n^{g}_{CO_2} = \frac{V_g P_{CO_2}}{Z_{CO_2} RT} \tag{3}$$

where V_g is the gas-phase volume in the autoclave corrected with the consideration of liquid volume change due to the solubility of CO_2. A high-pressure pycnometer was made in our laboratory with $^1/_{16}$ in. stainless steel tubing with high-pressure isolating valves. The pycnometer volume was measured at various temperatures and pressures, and gravimetric techniques were used to measure the density of loaded samples under high-pressure conditions of the experiments. The moles of CO_2 in the liquid phase $n^{l}_{CO_2}$ was then determined from

$$n^{l}_{CO_2} = n_{CO_2} - n^{g}_{CO_2} \tag{4}$$

The CO_2 loading in the liquid phase is defined as

$$\alpha_{CO_2} = \frac{n^{l}_{CO_2}}{n_{amine}} \tag{5}$$

where n_{amine} is the summation of the moles of MDEA and/or the other absorbents in the liquid phase. The uncertainty of $n^{l}_{CO}2$ is a direction function of uncertainty in measuring the exact volume of the reactor and density of liquid samples. The errors in measurement were estimated, and then the uncertainties in the moles of dissolved CO_2 were estimated using a standard technique.[15] Experiments were repeated at least three times, and accuracies better than ±0.5% were obtained in all volume and density measurement experiments. The solutions were prepared from deionized water. Reagent-grade MDEA was obtained from the Riedel-di Haën Company, Germany, with a purity of 98.5%. Piperazine (PZ) was obtained from the Merck Company with a purity of better than 99% and was used without further purification, and TMS (sulfolane) was obtained from the Fluka Company with a purity of 98% and was used as received.

Experimental Results and Discussion

Because of the low vapor pressure of pure MDEA, PZ, and TMS in the temperature range considered here, the partial pressure contributions of MDEA, PZ, and TMS in the vapor phase were neglected.[16]

To calibrate the experimental apparatus and to verify the accuracy of the measurements, the solubility of CO_2 in aqueous 2.00 kmol/m^3 MDEA at (25, 40, and 70) °C was measured and compared with the results presented by other researchers in Table 1. The results indicate good agreement between the experimental data obtained in this work and available literature data. The average absolute deviations (AADs) for all data points were less than 3.6%. AAD is defined as follows:

$$\mathrm{AAD} = \frac{\sum_{i=1}^{N} \left| \frac{P_{\mathrm{exptl},i} - P_{\mathrm{lit},i}}{P_{\mathrm{exptl},i}} \right|}{N} \qquad (6)$$

where P_{exptl} = experimental values, P_{lit} = literature values, and N = number of points.

Table 2 presents the numerical values of the solubility data. The results show that the absorption of CO_2 increases with PZ concentration, and this proves that PZ is an effective component in absorbing CO_2 into MDEA solutions. Temperature has a negative effect on CO_2 absorption. Table 3 lists the solubility of CO_2 in MDEA + TMS solutions for the same temperature range. The results show the solubility of CO_2 in MDEA + TMS solutions decreases with TMS concentration. Table 4 presents the solubility data for CO_2 dissolved in MDEA + PZ + TMS solutions. It is evident that the solubility of CO_2 decreases with TMS and increases with PZ and MDEA. The fact that CO_2 cannot be ionized in TMS (sulfolane) can explain the lower capacity of blends when TMS replaces water in the solutions.

Conclusions

The effects of the presence of PZ and TMS on the solubility of CO_2 in MDEA solutions were studied over a wide range of concentrations. The results show that PZ affects the solubility of CO_2 in MDEA solutions on the basis of the definition given for the CO_2 loading. TMS, however, has a negative effect on the solubility of CO_2 in MDEA solutions.

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