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Solubility of Carbon Dioxide in 30 mass % Monoethanolamine and 50 mass % Methyldiethanolamine Solutions

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The partial pressures of carbon dioxide (CO_2) over a 30 mass % aqueous solution of monoethanolamine (MEA) and a 50 mass % aqueous solution of methyldiethanolamine (MDEA) were measured. The range of partial pressures of CO_2 measured at 120 °C over 30 mass % MEA was from (7 to 192) kPa with loadings from (0.16 to 0.42). The partial pressures of CO_2 ranging from (66 to 813) kPa with loadings from (0.17 to 0.81) over 50 mass % MDEA were also measured at the temperatures (55, 70, and 85) °C. An approximate value of the enthalpy of solution of CO_2 in the aqueous MDEA solution was estimated using the solubility data.

Introduction

Aqueous solutions of alkanolamines are the most commonly used chemical absorbents for the removal of acidic gases from natural, refinery, and synthesis gas streams. Among them, aqueous monoethanolamine ($H_2NCH_2CH_2OH$, MEA) solutions have been used extensively for this purpose due to the rapid reaction rate, low cost of the solvent, ease of reclaiming, reasonable thermal stability, low molecular weight and thus high absorbing capacity on a mass basis, and relatively low solubility of hydrocarbons in the solution.[1−3] The disadvantages of aqueous MEA include the high enthalpy of reaction with CO_2 leading to higher desorber energy consumption, the formation of a stable carbamate and also the formation of degradation products with carbon oxysulfide (COS) or oxygen-bearing gases, inability to remove mercaptans, vaporization losses because of high vapor pressure, and a higher corrosiveness than that of many other alkanolamines.[2−4]

Methyldiethanolamine ($CH_3N(CH_2CH_2OH)_2$, MDEA) solutions are used for high pressure CO_2 removal and for selective removal of hydrogen sulfide (H_2S) from gas streams containing both CO_2 and H_2S. The use of MDEA solutions was first proposed by Frazier and Kohl.[5] The advantages of MDEA, a tertiary amine, over primary and secondary amines are, besides the selectivity for H_2S, a lower enthalpy of reaction with the acidic gases which leads to lower energy requirements for regeneration, a lower vapor pressure of the solution, a lower corrosiveness, and better thermal and chemical stability. The limitations of MDEA include a slower reaction rate with CO_2 and a lower absorption capacity at low concentrations of CO_2.[6,7]

A number of investigators have measured the solubility of CO_2 in 30 mass % MEA at temperatures from (0 to 150) °C with partial pressures of CO_2 ranging from 0.5 Pa to 20 MPa, as shown in Table 1. Among the investigators, only Goldman and Leibush,[10] Lee et al.,[12] and Jou et al.[2] measured the solubility of CO_2 in 30 mass % MEA at a regeneration temperature of 120 °C. However, there is a scarcity of data in the loading region most applicable for regenerator calculations, corresponding to partial pressures between (5 and 200) kPa. Moreover, the data of Lee et al.[12] have a consistent deviation of −0.04 mol of CO_2/mol of MEA compared to the data of Jou et al.[2]

The solubility of CO_2 in 50 mass % MDEA has also been determined by several investigators for temperatures between (25 and 200) °C with partial pressures of CO_2 ranging from 0.07 Pa to 6.6 MPa, as shown in Table 1. There is, however, a great scatter in the results, and no data exist for intermediate temperatures such as (55 and 85) °C.

The objective of the work described here is to obtain reliable data on the solubility of CO_2 in 30 mass % MEA at a regeneration temperature of 120 °C and 50 mass % MDEA at (55, 70, and 85) °C in the most interesting loading range which could serve as a standard for use in the modeling of vapor−liquid equilibria (VLE) in MEA and MDEA solutions.

Experimental Section

Sample MEA (purity >99 mass %) and MDEA (purity >98.5 mass %) solutions were prepared from Acros Organics without further purification and deionized water. The CO_2 (purity >99.99 mol %) and nitrogen (N_2) (purity >99.999 mol %) gases used were obtained from AGA Gas GmbH.

MEA Apparatus. The equilibrium measurements were carried out in a VLE apparatus with recirculation of the gas phase, as shown in Figure 1. The apparatus consists of three 300 cm^3 stainless steel cylinders (the equilibrium cells 1, 2, and 3) designed to operate at pressures up to 700 kPa and at temperatures up to 130 °C, a SERA (Seybert & Rahier GmbH) diaphragm pump (model ZR

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Table 1. Literature Review of 30 mass % MEA and 50 mass % MDEA Solubility Data

author	t/°C	p_{CO_2}/kPa
	30 mass % MEA	
Mason and Dodge (1936)[8]	0, 25, 50, 75	1.387−100.2
Lyudkovskaya and Leibush (1949)[9]	25, 50, 75	255.3−4124
Goldman and Leibush (1959)[10]	75, 100, 120, 140	0.5333−472.9
Lee et al. (1974)[11]	40, 100	1.151−6616
Lee et al. (1976)[12]	25, 40, 60, 80, 100, 120	0.2−6616
Nasir and Mather (1977)[13]	100	0.0005−0.52
Shen and Li (1992)[14]	40, 60, 80, 100	1.1−1975
Jou et al. (1995)[2]	0, 25, 40, 60, 80, 100, 120, 150	0.0012−19 954
	50 mass % MDEA	
Jou et al. (1982)[a] [15]	25, 40, 70, 100, 120	0.001 61−6570
Chakma and Meisen (1987)[a] [16]	100, 140, 160, 180, 200	138−4930
Austgen et al. (1991)[a] [17]	40	0.0102−93.6
Robinson (1993)[18]	40, 70, 100, 120	146.0−5327
Rho et al. (1997)[7]	50, 75, 100	0.775−140.3
Rogers et al. (1998)[19]	40	0.000 07−1.0018
Park and Sandall (2001)[20]	25, 50, 75, 100	0.78−140.4

[a] 48.8 mass %.

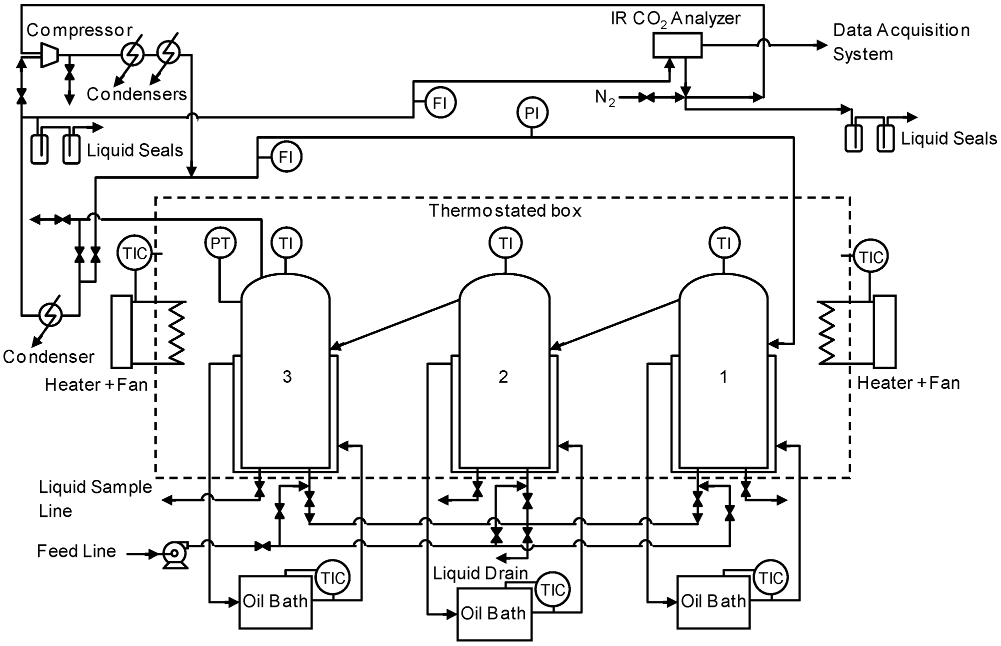

Figure 1. Equilibrium measurements for 30 mass % MEA.

408W), a KNF Neuberger compressor (model PM 15785-145), a Bourdon pressure gauge, a Druck PTX 610 pressure transducer with an accuracy of ±0.3% of full scale (800 kPa), four K-type thermocouples, and a Fisher-Rosemount nondispersive infrared (IR) CO_2 analyzer (model BINOS 100). The data acquisition uses FieldPoint FP-1000 and FP-AI-110.

Before starting the experiment, N_2 was flushed through the apparatus to purge the air within the cells. A preloaded 30 mass % MEA solution of 200 cm^3 was then fed into cell 1, while cells 2 and 3 held 150 cm^3 each. The cells placed in a thermostated box were heated by oil baths, and the temperatures were measured to within ±0.1 °C. To prevent boiling and vaporization of the solvent during the heating, the minimum initial pressure in the cells was set to 300 kPa. As the temperature reached the desired level (120 °C), the compressor increased the pressure up to 700 kPa and circulated the vapor. A backpressure valve was used to maintain the pressure at 700 kPa. Equilibrium was obtained when the temperature was established at a constant value and the CO_2 concentration in the vapor phase was constant. This took about (2 to 3) h including the heating-up period. After equilibrium was obtained, a liquid sample was withdrawn from cell 3 into a 75 cm^3 evacuated sampling cylinder such that the cylinder was completely filled by the liquid sample and then cooled to ambient temperature. The temperatures and pressures were automatically collected by the FieldPoint data acquisition system.

The vapor bleed extracted for composition measurement was cooled to 10 °C to condense water and MEA, and the CO_2 content was directly determined by IR analysis. The vapor phase in the IR analyzer, therefore, consisted of N_2, CO_2, and small amounts of H_2O and MEA. The concentration of CO_2 in the analyzer is then

$$y_{CO_2}^{IR} = \frac{n_{CO_2}^{IR}}{n_{CO_2}^{IR} + n_{N_2}^{IR} + n_{H_2O}^{IR} + n_{MEA}^{IR}} \tag{1}$$

where n denotes molar flow and the superscript IR denotes the vapor phase in the IR analyzer. Due to the low vapor pressure of MEA at 10 °C ($P_{MEA}^{\circ} = 0.012$ kPa),[21] MEA in the vapor phase through the analyzer could be disregarded.

The circulating vapor phase in the system at 120 °C consisted of N_2, CO_2, and significant amounts of H_2O and

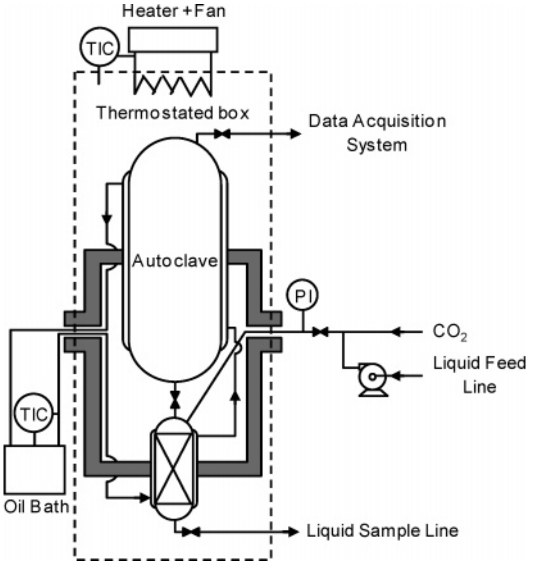

Figure 2. Equilibrium measurements for 50 mass % MDEA.

MEA. As noncondensable gases, the flows of CO_2 and N_2 were the same before and after the condenser. Equation 1 together with a mole balance will give the molar flow of CO_2 in the system

$$n_{CO_2} = y_{CO_2}^{IR}[n_T - (n_{H_2O} - n_{H_2O}^{IR}) - n_{MEA}] \qquad (2)$$

where n_T, n_{H_2O}, and n_{MEA} respectively denote the total moles and the moles of H_2O and MEA in the system. The partial pressure of CO_2 can then be calculated as

$$p_{CO_2} = y_{CO_2}^{IR}[P - (p_{H_2O} - p_{H_2O}^{IR}) - p_{MEA}] \qquad (3)$$

where P is the total pressure. The partial pressures of H_2O and MEA over the 30 mass % MEA solution were estimated using a Wilson equation.[22,23]

During the time of equilibration the cooling of the gas through the IR analyzer produced ~5 mL of condensed water containing small amounts of MEA. This water loss stems mainly from cell 1 where the gas enters and becomes saturated. The liquid losses of cells 2 and 3 were negligible, and the sample for liquid phase analysis was taken from cell 3. Separate heating for the cells was used to obtain accurate temperature control.

MDEA Apparatus. Figure 2 shows the equilibrium apparatus for MDEA in the shaking equilibrium cells. The apparatus consists of two connected autoclaves (1000 and 200 cm^3) which rotate 180° with 2 rpm and are designed to operate up to 2 MPa and 150 °C, Druck PTX 610 (max 800 kPa) and Schaevitz P 706-0025 (max 2.5 MPa) pressure transducers, and two K-type thermocouples. This apparatus was used for the MDEA tests, as the conditions ranged beyond those possible for the apparatus in Figure 1.

The autoclaves placed in a thermostated box were heated by an oil bath. During the heating-up period, the autoclaves were purged with CO_2 several times. The unloaded 50 mass % MDEA solution of 200 cm^3 was then injected into the smaller autoclave, and finally, CO_2 was injected to the desired pressure. Equilibrium was obtained when the temperature and pressure were constant to within ±0.2 °C and ±1 kPa. This took approximately (4 to 30) h. After equilibrium was obtained, a liquid sample was withdrawn from the smaller autoclave using a 75 cm^3 evacuated sampling cylinder where an unloaded MDEA solution of 25 cm^3 was injected into the cylinder before sampling. This was to ensure that all CO_2 in the liquid sample was totally absorbed. The cylinder was then cooled to ambient temperature. The partial pressure of CO_2 was measured by subtracting the partial pressures of H_2O and MDEA from the total pressure. As shown by Xu et al.,[24] it is reasonable to assume a Raoult's law behavior for the MDEA−water system. The data acquisition system used was FieldPoint.

Table 2. Solubility of CO_2 in 30 mass % MEA Solution at 120 °C

CO_2 loading (mol of CO_2/mol of MEA)	p_{CO_2}/kPa
0.1550	7.354
0.1766	9.314
0.1843	9.045
0.2085	15.51
0.2326	19.62
0.2381	25.20
0.2560	27.71
0.2901	39.18
0.2967	40.40
0.3004	43.49
0.3125	51.82
0.3191	58.57
0.3298	62.88
0.3424	77.59
0.3424	74.95
0.3500	83.61
0.3594	92.79
0.3882	137.9
0.4182	191.9

Liquid samples containing all bound CO_2 for both experiments were analyzed by the barium chloride ($BaCO_3$) method. The amount of hydrogen chloride (HCl) not used to dissolve $BaCO_3$ was titrated with 0.1 mol·L^{-1} sodium hydroxide (NaOH) by using an automatic titrator (Metrohm 702 SM Titrino) with an end point of pH 5.2. Due to solvent losses during the process at high operating temperature, the MEA concentrations were then determined by titration. A liquid sample of 0.5 cm^3 was diluted into deionized water of 75 cm^3 and titrated with 0.1 mol·L^{-1} sulfuric acid (H_2SO_4) using a Metrohm 702 SM Titrino instrument. The end point was obtained at pH 4 to 5.

Results and Discussion

Experimental solubility data for CO_2 in 30 mass % MEA solution were measured at 120 °C with an expanded uncertainty of ±0.5 °C and in 50 mass % MDEA solution at (55, 70, and 85) °C with an expanded uncertainty of ±0.2 °C. The uncertainties of the measured temperatures were estimated from the standard deviations and the half-width of temperature precisions (e.g., 0.05 °C for the MEA apparatus).[25] The experimental results are presented in Tables 2 and 3 and Figures 3 and 4.

The CO_2 loading analyses were performed by using two to five parallel liquid samples each titrated for CO_2 and MEA/MDEA contents. The relative standard uncertainty in the loadings estimated from the standard deviation of the loading measurements was ±2%.

The CO_2 partial pressure was measured online. The IR analyzer was calibrated using the calibration gases (0.5, 5, 10, and 20) mol % CO_2 with a relative standard uncertainty of ±2%. The analyzer readings at equilibrium varied within the half-width of its display, with the resolution being always < ±0.5%. The estimated relative expanded uncertainty in the CO_2 partial pressures was therefore found to be ±2%.

Goldman and Leibush,[10] Lee et al.,[12] and Jou et al.[2] have measured the equilibrium solubility for CO_2 in 30 mass %

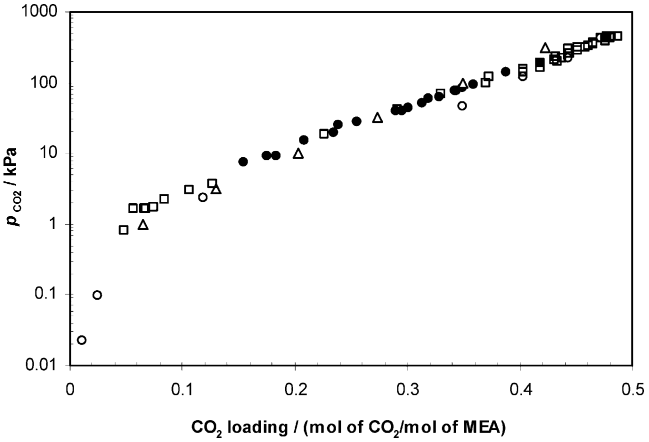

Figure 3. Comparison of the solubility of CO_2 in 30 mass % MEA solution at 120 °C: ●, experimental data; □, Goldman and Leibush;[10] △, Lee et al.;[12] ○, Jou et al.[2]

Table 3. Solubility of CO_2 in 50 mass % MDEA Solution at (55, 70, and 85) °C

CO_2 loading (mol of CO_2/mol of MDEA)	p_{CO_2}/kPa		
	55 °C	70 °C	85 °C
0.2758	65.75		
0.5325	172.8		
0.6411	277.4		
0.7126	388.8		
0.7387	490.5		
0.7402	485.1		
0.7479	492.3		
0.7825	585.1		
0.7971	684.9		
0.8133	779.8		
0.2367		95.70	
0.2790		117.7	
0.3582		177.2	
0.4029		220.9	
0.4718		273.6	
0.4834		306.7	
0.5259		379.1	
0.5489		430.4	
0.5858		486.7	
0.5894		488.3	
0.6058		581.4	
0.6609		688.1	
0.6786		776.9	
0.6898		813.4	
0.1658			129.7
0.1840			150.6
0.2609			242.9
0.3143			353.1
0.3269			355.6
0.3719			451.5
0.4112			555.7
0.4610			658.6
0.4887			754.9
0.4942			754.6

MEA solution at 120 °C. These are the only data found for this temperature. The data from this work are compared with their data. As seen from Figure 3, the equilibrium partial pressures of this work agree well with the data of Goldman and Leibush.[10] The data are also in good agreement with the smoothed data measurements of Lee et al.[12] at loadings from (0.26 to 0.36) and those of Jou et al.[2] for loadings above 0.36.

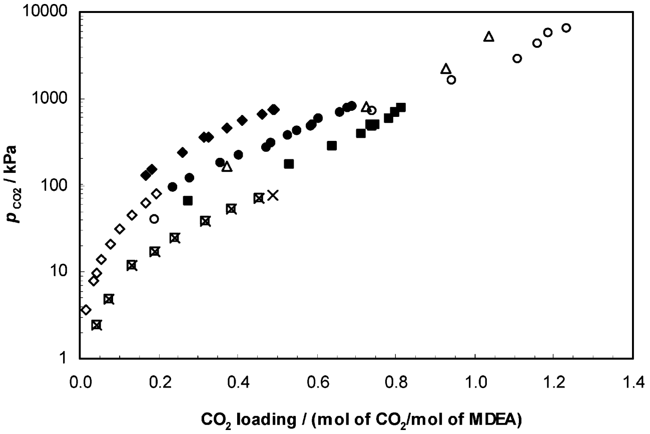

Figure 4. Comparison of the solubility of CO_2 in 50 mass % MDEA solution. Experimental data: ■, 55 °C; ●, 70 °C; ◆, 85 °C. Literature data: △, Robinson[18] (70 °C); ○, Jou et al.[15] (70 °C, 48.8 mass %); □, Rho et al.[7] (50 °C); ×, Park and Sandall[20] (50 °C); ◇, Rho et al.[7] and Park and Sandall[20] (75 °C).

Literature data for comparison of the equilibrium solubility of CO_2 in 50 mass % MDEA solution at the temperatures used here are limited. Only at 70 °C could the data directly be compared to those of Robinson.[18] The data were also compared to those of Jou et al.,[15] Rho et al.,[7] and Park and Sandall.[20] At 70 °C, the measured CO_2 partial pressures from this work are slightly higher than those of Robinson.[18] The data are also higher than those of Jou et al.[15] This is natural, as the concentration of MDEA used in this work is slightly higher than that used by Jou et al.[15] (48.8 mass %). Rho et al.[7] found that the higher the concentration of amine used, the higher the partial pressure of CO_2 measured at a fixed temperature and CO_2 loading. The equilibrium partial pressures of CO_2 at (55 and 70) °C were then compared to those of Rho et al.[7] and those of Park and Sandall[20] but at (50 and 75) °C. The measured data at 55 °C show higher partial pressures of CO_2 compared to those of Rho et al.[7] and those of Park and Sandall[20] at 50 °C. This is natural in view of the temperature difference. Extrapolated data of Rho et al.[7] and of Park and Sandall[20] at 75 °C tend to predict higher partial pressures of CO_2 at higher CO_2 loadings compared to this work for a temperature of 70 °C. This is because the temperature used in this work was 5 °C lower than in the work of Rho et al.[7] and that of Park and Sandall.[20]

An approximate value of the differential enthalpy of solution of CO_2 in the aqueous 50 mass % MDEA solution in such a loading was calculated by use of the following form of the Gibbs−Helmholtz equation

$$\frac{\Delta H_s}{R} = \left(\frac{\partial \ln p_1}{\partial (1/T)}\right)_{x_1} \tag{4}$$

where the subscript 1 refers to CO_2 and x_1 is the mole fraction or equilibrium loading of CO_2.

The enthalpy of solution is dependent on the loading. The value of ΔH_s in 50 mass % MDEA solution at a loading of 0.50 was found to be 53.4 kJ·mol^{-1} of CO_2. The standard uncertainty for this value was estimated to be ±5%. This value agrees well with the value 53.2 kJ·mol^{-1} of CO_2 (at 48.8 mass % MDEA) at a loading of 0.50 which was proposed by Jou et al.[15] but is higher than the value 30 kJ·mol^{-1} of CO_2 (30 mass % MDEA) at a loading of 0.51 reported by Mathonat et al.[26]

Conclusions

The vapor−liquid equilibrium data of CO_2 in 30 mass % MEA solution and in 50 mass % MDEA solution were measured at 120 °C and at (55, 70, and 85) °C, respectively. In addition to the literature data, the VLE data of MEA at stripping conditions (120 °C) should be useful for the desorber design using MEA and those of MDEA should be useful for the design of absorption columns using MDEA to remove CO_2. Both the VLE data of MEA and those of MDEA could also serve as a standard for use in the modeling of VLE in MEA and MDEA solutions. Last, the enthalpy of solution of CO_2 in the aqueous 50 mass % MDEA solution at a loading of 0.50 was found to be 53.4 kJ·mol^{-1} of CO_2.

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