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Solubility of Carbon Dioxide in Water and Aqueous Solution Containing Sodium Hydroxide at Temperatures from (293.15 to 393.15) K and Pressure up to 5 MPa: Experimental Measurements

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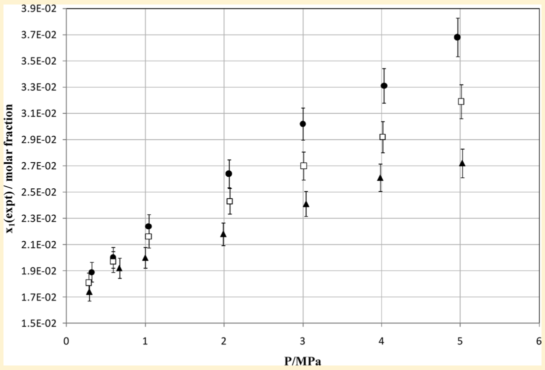
ABSTRACT: New experimental solubility data for carbon dioxide in pure water and in aqueous solution containing sodium hydroxide in the temperature range (293.15 to 393.15) K and pressures up to 5 MPa are presented in this work. A new experimental apparatus based on an equilibrium cell with a constant volume allowing the measurement of carbon dioxide solubility is presented. The liquid phase composition at the thermodynamic equilibrium is determined by an analytical method (ion chromatography) and a static synthetic method. The CO_2 + water system is used to validate the experimental apparatus; therefore data obtained are compared with literature data.



INTRODUCTION

It is well-known that the production of carbon dioxide from burning fossil fuel participates in the global warming. This issue generates a growing interest for CO_2 capture and storage from postcombustion or oxy-fuel combustion. The design and the optimization of separation processes require a good knowledge of thermodynamics properties of phase equilibria. This paper presents a new apparatus for determining the gas solubility in aqueous solutions. The device was set up to allow the study of the solubility of gas mixtures involved in CO_2 capture and storage processes (i.e., CO_2, O_2, NO_x, SO_2) in aqueous solution. The different gas analyzers need a certain gas volume to work; thus, the equilibrium cell used in this work was designed with a large internal volume. The first step in this study is the experimental equipment validation with the CO_2 + water system in the temperature range (298.15 to 393.15) K and up to 5 MPa. Then the CO_2 + NaOH + water system is investigated in concentration of 1 mol·kg^{-1} of water, in the (293.15 to 333.15) K temperature range and up to 5 MPa. First a literature review is presented on the CO_2 + water system and on the CO_2 + NaOH + water system; then the experimental apparatus, the operating procedure, and analysis methods are described, and last, the results obtained are presented.

LITERATURE REVIEW

CO_2 + Water System. There is a large number of experimental data available in scientific literature concerning the solubility of carbon dioxide in pure water. Four reviews about this system have been published since 2003. Diamond and Akinfiev[1] evaluated literature data in the temperature range from (271.15 to 373.15) K and from (0.1 to 100) MPa. Spycher and Pruess[2] reviewed the mutual solubilities of CO_2 and water in the temperature range from (285.15 to 373.15) K and up to 60 MPa. Chapoy et al.[3] reviewed reliable solubility data of CO_2 in water at (273.15 to 373.15) K and at (0.07 to 70.9) MPa. In 2007 Ji et al.[4] reviewed solubility data and water content in the vapor phase for the CO_2 + water system in wider temperature and pressure ranges—that is, (0 to 473.15) K and (0.07 to 70.9) MPa. Thus the studies with data at T = (298.15, 323.15, 348.15, 373.15, and 393.15) K and the pressure range of interest were selected and reported in Table 1.

This table summarizes for each study the temperature and pressure ranges of experiments. Two types of equilibrium cell are used: a variable volume cell as that used by Nighswander et al.[5] and Chapoy et al.,[3] or a constant volume cell (Dalmolin et al.,[6] Valtz and Chapoy,[7] Bamberger et al.,[8] Matous et al.,[9] Kiepe et al.[10]). Koschel et al.[11] developed a calorimetric technique to determine enthalpic and solubility data. Bermejo et al.[12] used a Cailletet apparatus for gas solubility measurements. The volume of the cell is generally about a few dozen cubic centimeters.

Several authors determined the amount of CO_2 transferred in the aqueous phase by taking a liquid sample, degassing at atmospheric pressure, and measuring the volume of exsolved CO_2, such as Zelwinskii,[13] Wiebe and Gaddy,[14] and Gillepsie and Wilson.[15] Bamberger et al.,[8] Nighswander et al.,[5] and

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Table 1. Literature Data for the Carbon Dioxide–Water System

author	temperature/K	pressure/MPa
Dalmolin et al.[6]	288.0 to 323.0	0.092 to 0.473
Zelwinskii[13]	273.0 to 373.0	1.1 to 9.4
Wiebe and Gaddy[14]	323.2 to 373.2	2 to 71
Valtz and Chapoy[7]	278.2 to 318.2	0.465 to 7.963
Chapoy et al.[3]	273.2 to 373.2	0.190 to 9.333
Bermejo et al.[12]	296.7 to 369.7	1.55 to 8.34
Koschel et al.[11]	323.1 to 373.1	2 to 20
Matous et al.[9]	303.0 to 353.0	1 to 3.9
Bamberger et al.[8]	313.0 to 353.0	1 to 14
Müller et al.[16]	373.15 to 473.15	0.3 to 2.3
Kiepe et al.[10]	313.2 to 393.2	0.01 to 9.26
Gillepsie and Wilson[15]	298.0 to 366.0	0.7 to 20
Nighswander et al.[5]	353.0 to 471.0	2.04 to 10.2

Matous et al.[9] used this analysis method with a water trapping. Valtz and Chapoy[7] used gas chromatography as an analysis method. Müller et al. also used gas chromatography but only to measure the gas composition.[16] Chapoy et al.[3] introduced a known amount of CO_2 and water in the cell and detected the bubble point. Dalmolin et al.[6] calculated the amount of CO_2 dissolved in pure water through pressure drop measurements. Kiepe et al.[10] used a static synthetic method based on an isothermal and isochoric flash calculation.

CO_2 + NaOH + Water System. For the CO_2 + NaOH + water system, data are scarce. Rumpf et al.[17] studied in 1998 the solubility of carbon dioxide in NaOH aqueous solution in the (313 to 433) K temperature range and at pressures up to 10 MPa. The equipment used was a constant volume cell (30 cm^3). A known amount of CO_2 is introduced in the cell, and then the solvent is introduced until the gas is completely dissolved. Then the pressure is decreased step by step by withdrawing amounts of liquid phase until the first bubble appears. This study investigated three NaOH concentrations: 0.9569, 0.9613, and 1.0441 moles of sodium hydroxide per kilogram of water.

EXPERIMENTAL SECTION

Chemicals. Carbon dioxide is provided by Air Liquide with a certified purity of 99.7 % (CAS Registry No. 124-38-9). Water is purified by a Barnstead Easypure RoDi with a resistivity of 18.2 Ω. Sodium carbonate (CAS Registry No. 497-19-8) for the calibration curve is provided by VWR with a certified purity of 99 %. Sodium hydroxide (CAS Regsitry No. 1310-73-2) is provided by VWR with a certified purity of 99 %.

Apparatus. Solubility measurements were performed with an apparatus through a static method. This apparatus (see Figure 1) is based on a Hastelloy C276 well-stirred equilibrium cell with a double jacket; its volume is 2400 cm^3, and it can be operated up to 6 MPa from (293 to 393) K. The temperature is strictly controlled and regulated by a thermostatted bath. Because of the large volume cell, a thermal gradient could exist; on the top of the apparatus a heating resistance allows us to minimize this thermal gradient. Moreover, the temperature is measured at three locations: in the vapor phase, at the vapor–liquid interface, and in the liquid phase through three 100 Ω platinum probes. These three thermometers were calibrated by the Laboratoire National d'Essais (Paris), and the resulting uncertainty is less than 0.06 K. The pressure is measured by a pressure transducer (PA-25) from Keller; its accuracy is ± 0.01 MPa. Two sapphire windows enable observation of the different phases. A first mass flowmeter (FT01) on the gas loading line has a volumetric counter allowing the measurement of gas volume loaded in the cell. A second mass flowmeter (FT02) allows gas flow control in the gas phase analyzer. The stirrer, a Rushton turbine in the liquid phase and a four-blade impeller in the gas phase, ensures the homogeneity of the two phases. Safety devices like a rupture disk and agitation cutoff are also provided.

Generally, equilibrium cells have an internal volume of one to a few dozen cubic centimeters. The apparatus described have

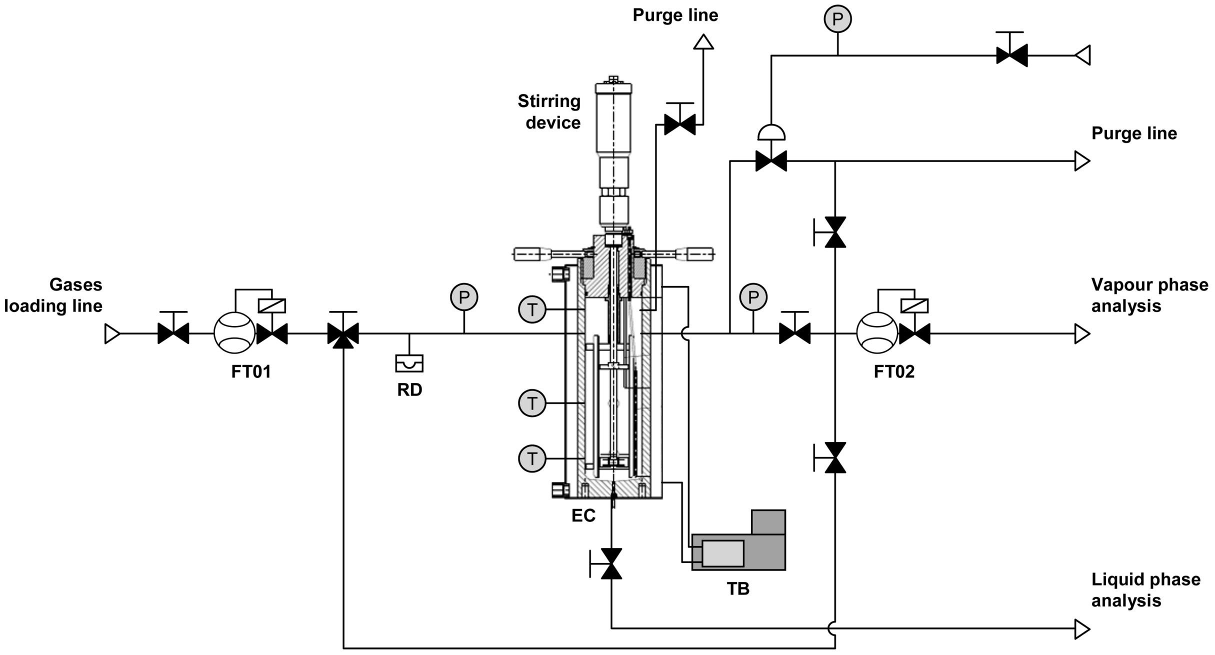

Figure 1. Scheme of the experimental equipment: FT01, FT02: mass flowmeters; RD: rupture disk; EC: equilibrium cell; TB: thermostatted bath; P: pressure transducer or manometers; T: temperature probes.

an equilibrium cell with an unusually large volume. As said before the equipment was designed to allow the study of gas mixtures ($CO_2/O_2/NO_x/SO_x$) solubility in aqueous solutions. The gas analyzer of NO_x and SO_2 requires a large amount of gas to work. The choice of this internal volume is justified by the need to analyze both aqueous and gaseous phases without disturbing the thermodynamic equilibrium during sample withdrawal. However the scope of this study is the solubility of carbon dioxide in aqueous solution containing sodium hydroxide, so there are no other impurities than water in the gas phase.

Operating Procedure. The equilibrium cell and lines are purged using a vacuum pump prior to introduce the aqueous solution. The mass of solvent loaded in the cell—about 500 g—is weighted using an analytical balance whose absolute accuracy is 0.1 g. Then the gas is introduced up to the desired pressure. Thanks to the mass flowmeter FT01, the amount of gas loaded in the cell is known with a relative accuracy of 1 %. The thermostatted bath temperature and the heated resistance temperature are monitored to work at the desired temperature in the cell. Then the stirring is started; 700 rpm is enough for gas aspiration through the stirrer, and then the contact area between the two phases is sharply increased. The apparatus was first used to determine the necessary time to obtain the equilibrium. Once the cell is loaded, samples are analyzed by ion chromatography until carbon dioxide concentration in water no longer varies. It appears that the thermodynamic equilibrium is achieved in one hour for both the CO_2/water system and the CO_2/NaOH/water system. Figure 2 shows pressure and CO_2 concentration evolution according to time.

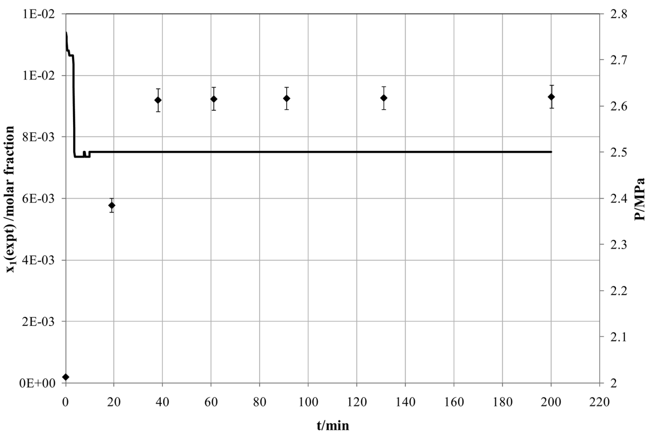

Figure 2. Molar fraction of carbon dioxide in the aqueous phase and total pressure evolution according to time at T = 313.15 K.

When the thermodynamic equilibrium is achieved, three samples of the liquid phase are analyzed by ion chromatography. No pressure variation due to the withdrawal of the three samples is observed because the taken volume (purge and samples) is very small—less than 5 mL—compared to the total volume of liquid in the equilibrium cell.

Analysis. The composition of the aqueous phase is determined by two methods: direct analysis of the aqueous phase by ion chromatography and static synthetic method based on an assessment of the amount of carbon dioxide transferred in the aqueous phase calculated by mass balance.

Principle of Ion Chromatography Analysis. Ion chromatography is an analytical method for determining the amount of ion in an aqueous solution. The liquid chromatography system used in this study consists in a Dionex ICS-900 ion chromatography equipped with an ICE-AS1 column (9 × 250 mm). This column is specific to weak acid anion analysis. Data were processed using Chromeleon software. Samples are carried along and diluted by the elution thanks to a pump; then they pass through the column. Several samples (obtained from aqueous solution of $NaHCO_3$, Na_2CO_3, and K_2CO_3) with a known concentration in total CO_2 are tested, and whatever the sample, the ion chromatography allows the quantification of total CO_2. Calibration curves are obtained from aqueous solutions of Na_2CO_3 analysis. The maximum volume of the sample introduced in the injection valve is 2 μL. The chromatograph is directly connected to the equilibrium cell. Samples are pushed in the injection valve thanks to the pressure in the cell.

Balance Method. In addition to analysis by ion chromatography, the amount of carbon dioxide dissolved in the liquid phase is calculated. Indeed, the initial amount of gas loaded in the cell is known thanks to the gas flowmeter FT01 as well as the introduced mass of aqueous phase. At thermodynamic equilibrium, it is possible to calculate the amount of CO_2 in the gas phase from the pressure, the temperature, the gas volume, and a compressibility factor obtained in the National Institute of Standards and Technology (NIST) database.[18] The difference between initial amount and final amount of CO_2 at the equilibrium gives the quantity transferred in the aqueous phase.

Experimental Uncertainties. The estimation of measurement uncertainties is based on the *Guide to the Expression of Uncertainty in Measurement.*[19] The uncertainty obtained for the ion chromatography is about 4 % according to this method. The calculation for balance method gives an uncertainty of about 17 % for the CO_2 + water system and between 5 and 16 % (function of CO_2 molar fraction) for the CO_2 + NaOH + water system. This last result is much higher than the uncertainty of the ion chromatography method because each step of the method contributes to increase the overall uncertainty (pressure, temperature, gas flowmeter, cell volume). This method for calculating uncertainties is based on theoretical considerations.

The calculation of uncertainties based on reproducibility experiment with the single factor ANOVA (analysis of variance) method gives an average uncertainty of 3.4 % for ion chromatography. The experimental average uncertainty for the balance method is 12 %, but this uncertainty is very variable. The coverage factor chosen to obtain a level of confidence of 95 % is 4.3 according the Student's distribution.

This assessment of uncertainties enabled us to choose the most accurate method, that is, chromatography analysis. Consequently, all results presented in this work are obtained from ion chromatography analysis.

■ RESULTS AND DISCUSSION

CO_2 + Water System. Five isotherms in the temperature range from (293.15 to 393.15) K were investigated. These solubility data are plotted in Figures 3 to 7 for T = (298.15, 323.15, 348.15, 373.15, and 393.15) K, respectively. Literature data available in the same temperature and pressure conditions are also reported in these figures. The experimental results for the solubility of carbon dioxide in pure water are given in Table 2.

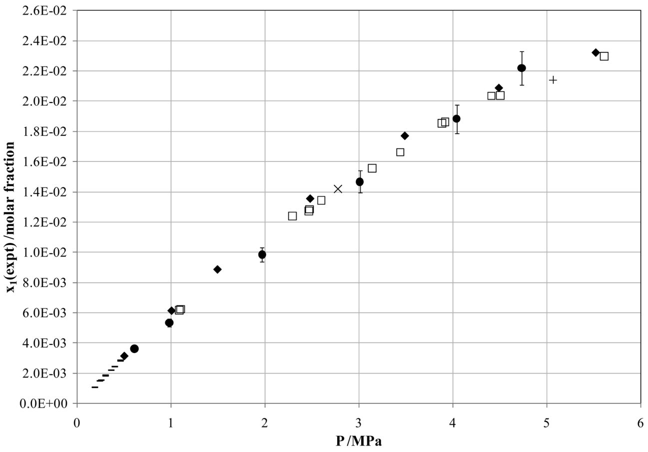

Figure 3. Solubility of carbon dioxide (1) in pure water (2) at $T = 298.15$ K: ●, this work; ◆, Valtz and Chapoy;[7] □, Zelwinskii et al.;[13] ×, Chapoy et al.;[3] —, Dalmolin et al.;[6] +, Wiebe and Gaddy.[14]

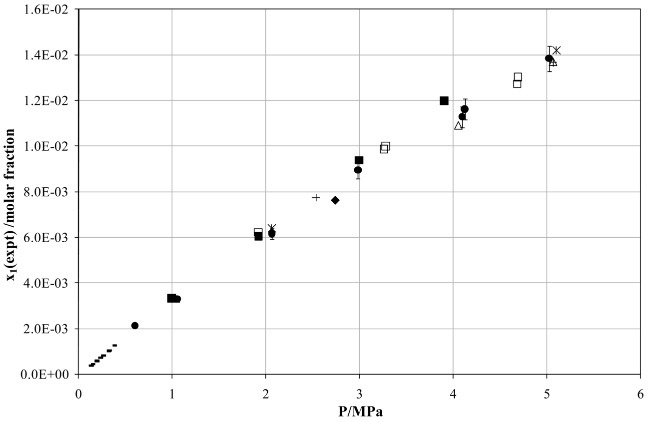

Figure 4. Solubility of carbon dioxide (1) in pure water (2) at $T = 323.15$ K: ●, this work; ◆, Bermejo et al.;[12] □, Zelwinskii et al.;[13] —, Dalmolin et al.;[6] +, Wiebe and Gaddy;[14] ■, Matous et al.;[9] △, Bamberger et al.;[8] ∗, Koschel et al.[11]

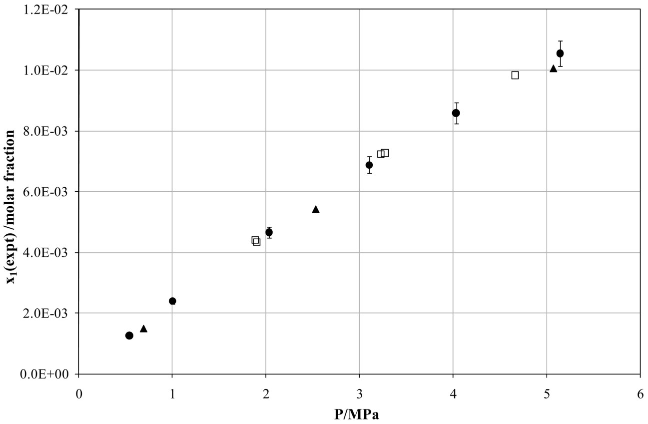

Figure 5. Solubility of carbon dioxide (1) in pure water (2) at $T = 348.15$ K: ●, this work; □, Zelwinskii et al.;[13] ▲, Gillepsie and Wilson.[15]

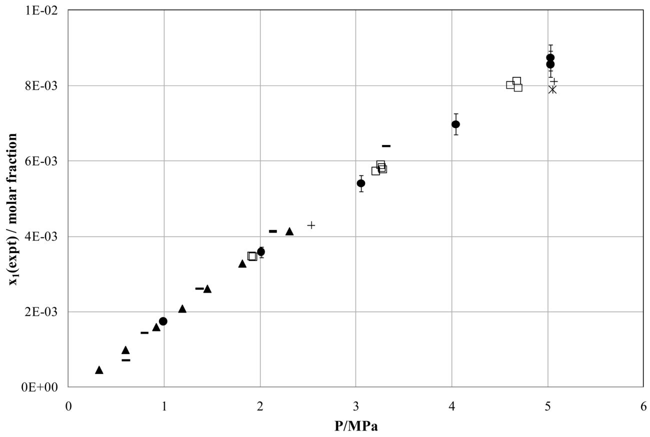

Figure 6. Solubility of carbon dioxide (1) in pure water (2) at $T = 373.15$ K: ●, this work; ▲, Müller et al.;[16] □, Zelwinskii et al.;[13] —, Kiepe et al.;[10] +, Wiebe and Gaddy;[14] ∗, Koschel et al.[11]

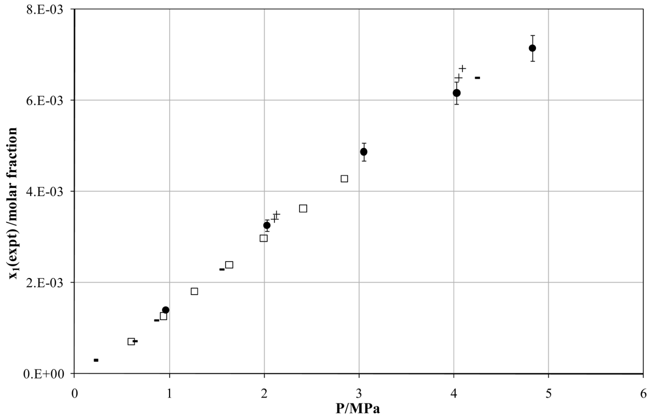

Figure 7. Solubility of carbon dioxide (1) in pure water (2) at $T = 393.15$ K: ●, this work; □, Müller et al.;[16] —, Kiepe et al.;[10] +, Nighswander et al.[5]

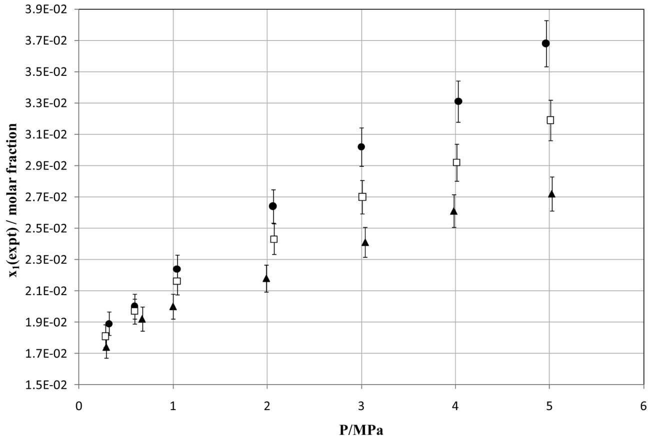

Figure 8. Solubility of carbon dioxide (1) in aqueous solutions containing sodium hydroxide (3) at 1 mol of sodium hydroxide/kg of water (2) at T = ●, 293.15 K; □, 313.15 K; ▲, 333.15 K; this work.

As expected the solubility of carbon dioxide in pure water increases with increasing pressure and decreases with increasing temperature.

The five graphs show a good agreement with literature data. The data obtained in this work are presented with an uncertainty of 4 % corresponding to the ion chromatography uncertainty. Experimental data obtained with the new equipment have a deviation with literature data less than 4 %. Then the new cell with large internal volume is adapted for the measurement of vapor−liquid equilibria. Results show that the operating procedure and the analysis method can be validated.

Table 2. Experimental Results for Carbon Dioxide (1) Solubility in Pure Water (2)

T = 298.15 K ± 0.06 K		T = 323.15 K ± 0.06 K	
P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %	P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %
0.61	0.00362	0.6	0.00214
0.98	0.00534	1.05	0.00331
1.97	0.00985	2.06	0.00616
3.01	0.0147	2.98	0.00895
4.04	0.0188	4.09	0.0113
4.73	0.0222	4.12	0.0116
		5.02	0.0139
T = 348.15 K ± 0.06 K		T = 373.15 K ± 0.06 K	
P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %	P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %
0.54	0.00126	0.99	0.00175
1	0.00241	2.01	0.00359
2.03	0.00467	3.05	0.00541
3.1	0.00688	4.04	0.00697
4.03	0.00859	5.03	0.00874
5.14	0.0106	5.03	0.00857

T = 393.15 K ± 0.06 K	
P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %
0.96	0.00141
2.03	0.00326
3.05	0.00487
4.03	0.00617
4.83	0.00715

CO_2 + NaOH + Water System (1 mol of NaOH/kg of Water). Three isotherms at T = (293.15, 313.15, and 333.15) K are investigated in the (0.2 to 5) MPa pressure range. Results obtained are plotted in Figures 8 and 10 given in Table 3. As in the carbon dioxide–water system, the solubility decreases with increasing temperature and increases with increasing pressure.

Table 3. Experimental Results for Carbon Dioxide (1) Solubility in Aqueous Solution Containing Sodium Hydroxide (3) at 1 mol of Sodium Hydroxide/kg of Water (2)

T = 293.15 K ± 0.06 K		T = 313.15 K ± 0.06 K	
P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %	P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %
0.32	0.0189	0.28	0.0181
0.59	0.0200	0.59	0.0197
1.04	0.0224	1.04	0.0216
2.06	0.0264	2.07	0.0243
3	0.0302	3.01	0.0270
4.03	0.0331	4.01	0.0292
4.96	0.0368	5.01	0.0319

T = 333.15 K ± 0.06 K	
P(expt)/MPa ± 0.01	x_1(expt) /molar fraction ± 4 %
0.29	0.0174
0.67	0.0192
1.00	0.0200
1.99	0.0218
3.04	0.0241
3.98	0.0261
5.02	0.0272

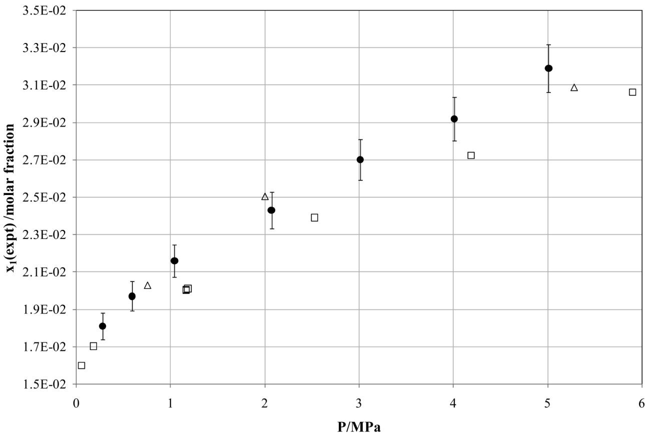

Figure 9. Solubility of CO_2 (1) in an aqueous solution of NaOH at T = 313.15 K: comparison with Rumpf et al. Data:[17] ●, this work; □, Rumpf et al.[17] 0.9613 mol·kg^{-1}; △, Rumpf et al.[17] 1.0441 mol·kg^{-1}.

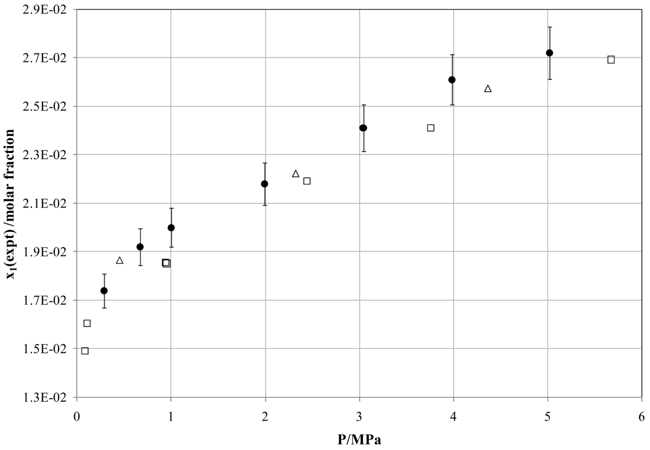

Figure 10. Solubility of CO_2 (1) in an aqueous solution of NaOH at T = 333.15 K: comparison with Rumpf et al. Data:[17] ●, this work; □, Rumpf et al.[17] 0.9613 mol of NaOH/kg; △, Rumpf et al.[17] 1.0441 mol of NaOH/kg.

Data obtained in this work are compared to Rumpf et al. data[17] in Figures 9 and 10 at T = (313.15 to 333.15) K. It must be noted that we do not know the uncertainty of the molality of carbon dioxide in the aqueous solution obtained by Rumpf et al.[17] Indeed Rumpf et al.[17] only give the uncertainties for the measurements of pressure, temperature, and molality of sodium hydroxide in the aqueous solution.

These figures show that data obtained in this work are consistent with Rumpf et al.[17] data up to 2 MPa. For higher pressures a deviation is observed: the molar fractions of CO_2 in the liquid phase are slightly higher than Rumpf et al.[17] data.

A reproducibility study was performed, and all data obtained for the same pressure have a 4 % uncertainty. Moreover data obtained by balance are in a good agreement with data obtained by ion chromatography; there is about 1 % deviation between the two methods.

Data obtained at T = 293.15 K cannot be compared to literature because Rumpf et al.[17] did not investigate this temperature, and there are no other studies about the solubility of carbon dioxide in aqueous solutions of sodium hydroxide in the open literature.

CONCLUSIONS

A new apparatus was built and set up for the determination of vapor−liquid equilibrium. New experimental data on the solubility of carbon dioxide in pure water were performed in the (298.15 to 393.15) K temperature range and up to 5 MPa. Two analysis methods are possible, but the expression of uncertainties shows that the more accurate results are given by ion chromatography analysis. These data were compared to literature data and showed a very good agreement in the whole temperature and pressure ranges. Then the new cell equilibrium and experimental procedure were validated.

This device was used to investigate CO_2 solubility in aqueous solution containing 1 $mol \cdot kg^{-1}$ of water of sodium hydroxide. There is a lack of experimental data for this system, especially for the low temperatures. The three isotherms are performed at T = (293.15, 313.15, and 333.15) K up to 5 MPa.

The experimental equipment will be used to investigate other isotherms in the same temperature and pressure range but with other concentrations in sodium hydroxide. Then, the cosolubility of binary gas mixtures in pure water and sodium hydroxide will be studied.

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