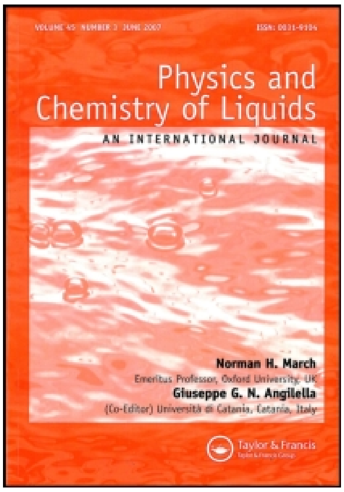

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Solubility of dilute SO_2 and CO_2 in dimethyl sulfoxide

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The solubility of SO_2 and CO_2 in dimethyl sulfoxide has been determined in the temperature range from 293.15 to 313.15 K and partial pressure of SO_2 from 0.15 to 2.62 kPa, and the partial pressure of CO_2 from 5 to 18 kPa. A solubility model is proposed and the solubilities calculated by the model show good agreement with the experimental data.

Keywords: Solubility; Gas–liquid equilibrium; Sulfur dioxide; Carbon dioxide; Model

1. Introduction

An area of major interest in recent years is the control of sulfur dioxide emissions from power plants and industrial plants. Flue gases often contain dilute sulfur dioxide and it is necessary to remove these acidic components. Many of the processes involve the absorption of sulfur dioxide into an alkaline solution and a chemical reaction with an amine. An alternate purification process is physical absorption with polar organic solvents. Physical absorption with a polar solvent provides a convenient and economic method for removing acid gases [1–3]. In designing an absorption system to purify flue gases, it is necessary to know the solubilities of SO_2 and CO_2 in the absorbing solvents, but the fundamental data that have been published for dimethyl sulfoxide (DMSO) are very limited [1,4–6]. It is necessary to have solubility data of SO_2 and CO_2 in the absorbing solvents based on the need of industrial design. This work reports solubility data suitable for possible absorption processes. In the present study, the solubilities of sulfur dioxide and carbon dioxide in DMSO at temperatures from 293.15 to 313.15 K, and partial pressures of SO_2 from 0.15 to 2.62 kPa, and the partial pressure of CO_2 from 5 to 18 kPa have been measured. A solubility model is proposed and the solubilities that have been calculated by the model agree with the experimental data.

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2. Experimental

2.1. *Materials and analysis*

Dimethyl sulfoxide was of AR grade and its purity greater than 99%. It was degassed under vacuum. Water was purified by vacuum distillation and deionization. The dilute SO_2 mixture was prepared by mixing sulfur dioxide and nitrogen. The sulfur dioxide, carbon dioxide and nitrogen had a purity $x(SO_2) > 0.999$, $x(CO_2) > 0.999$ and $x(N_2) > 0.99999$, respectively. The total solubility of SO_2 and CO_2 was measured using the apparatus shown in figure 1 [7]. A SO_2 analyser with SO_2 sensor (type NTS 100, Nanjing, China) was used for the SO_2 analysis ($\leq 0.5\%$ SO_2) of the gas phase. The uncertainty of the SO_2 analyser was ± 10 ppm; more than 0.5% SO_2 concentration was analysed by iodometry. The two methods were calibrated before measuring the SO_2 concentration. A CO_2 analyser (type RD-7AG, Nanjing, China) was used for the CO_2 analysis of the gas phase. The uncertainty of the CO_2 analyser was $\pm 3\%$ of the maximum measured value. The measurement range of the CO_2 analyser is 0–20%. The SO_2 in the liquid was analysed by potassium permanganate titration. The solubility of CO_2 in the liquid was calculated by the total solubilities of SO_2 and CO_2 minus the solubility of SO_2.

2.2. *Solubility measurement*

The solubility of SO_2 and CO_2 in DMSO was measured using the apparatus shown in figure 1.

The principle for this method is to bring a known volume of liquid into contact with a gas in a closed system at a constant temperature and pressure. Equilibrium is reached by agitating the liquid via an external magnetic stirrer for some time until no change in the volume of the gas is observed. The amount of gas absorbed is measured volumetrically; typical volumes were from 2 to 40 cm^3, with an uncertainty of ± 0.05 cm^3.

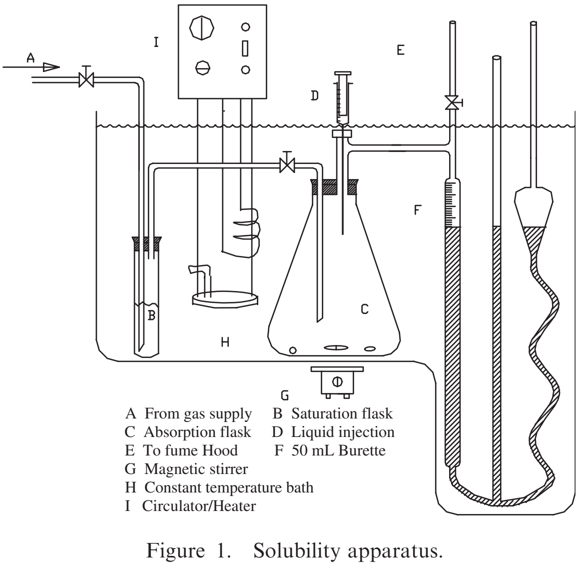

Figure 1. Solubility apparatus.

The procedure for making a solubility measurement is described now. A gas saturated with the vapors of the absorbing liquid was passed through the system at a constant temperature long enough to completely purge the absorption flask. Then the inlet and outlet valves were closed. The height of the mercury in the three branches was leveled, ensuring that the pressure in the flask was atmospheric, and the position of the meniscus was recorded. An aliquot of degassed liquid that was kept at the same temperature as that of the experiments was weighed, given an approximate mass and injected into the absorption flask. The uncertainty of the mass was ±0.00001 g. The liquid sample was continuously agitated via an external magnetic stirrer. The height of the mercury in the three branches was leveled every few minutes to make sure that the gas phase was at an atmospheric pressure. Equilibrium was reached when the height of the mercury in each branch leveled and remained constant. The measured volume change was equal to the volume of liquid sample minus the volume of the gas absorbed. The whole apparatus was kept at a constant temperature inside a temperature-controlled bath. The uncertainty of temperature was ±0.05 K.

2.3. *Data reduction*

The solubility of SO_2 and CO_2 and its equilibrium composition of gas phase are calculated from material balance and equations of equilibrium for each component. Typically, there are two phases in the system: (1) The gas phase which includes SO_2, CO_2, N_2 and solvent vapor; (2) The liquid phase which includes solvent and the dissolved sulfur dioxide and carbon dioxide.

The total moles in the gas phase in the cell (n_{inlet}/mol) can be calculated from the gas phase analysis and modified SRK equations of state [8].

The inlet SO_2, CO_2 and N_2 moles in the gas phase in the cell ($n_{SO_2,1}$/mol) can be calculated from equations (1)–(3)

$$n_{SO_2,1} = y_{SO_2,1} \times n_{\text{inlet}} \tag{1}$$

$$n_{CO_2,1} = y_{CO_2,1} \times n_{\text{inlet}} \tag{2}$$

$$n_{N_2,1} = (1 - y_{SO_2,1} - y_{CO_2,1}) \times n_{\text{inlet}} \tag{3}$$

where $y_{SO_2,1}$, $y_{CO_2,1}$ is the inlet mole fraction of SO_2, CO_2, it can be calculated from inlet concentration of SO_2, CO_2 in the gas phase, and n_{inlet} is the inlet total moles of components.

The total volume of the SO_2, CO_2 gas absorbed can be calculated from the measured volume change of the burette and injected mass of the liquid sample. The moles of dissolved SO_2, CO_2 (n_{SO_2}/mol kg^{-1}) are calculated as follows:

$$n_{SO_2+CO_2}/(\text{mol kg}^{-1}) = \left(\frac{m/\text{g}}{1.1195 - 0.001\,(T/\text{K} - 273.15)} - \Delta V/\text{cm}^3\right) \tag{4}$$

where n_{SO_2} is the SO_2 moles solubility; m represents injected mass of the liquid sample; T is the absolute temperature; ΔV is the measured volume change of burette.

The solubility of SO_2 can be obtained by potassium permanganate titration, the solubility of CO_2 can be calculated by the total solubility of SO_2 and CO_2 minus the solubility of SO_2.

The equilibrium SO_2 moles in the gas phase ($n_{SO_2,2}$/mol) are equal to the inlet SO_2 moles minus the dissolved SO_2 moles.

$$n_{SO_2,2} = n_{SO_2,1} - n_{SO_2} \times c_1 \tag{5}$$

where $n_{SO_2,1}$ is the inlet SO_2 moles, n_{SO_2} is SO_2 moles solubility in $\mathrm{mol\,kg^{-1}}$ and c_1 is the unit conversion coefficient.

The equilibrium CO_2 moles in the gas phase ($n_{CO_2,2}$/mol) are equal to the inlet CO_2 moles minus the dissolved CO_2 moles.

$$n_{CO_2,2} = n_{CO_2,1} - n_{CO_2} \times c_1 \tag{6}$$

The equilibrium DMSO moles in the gas phase can be calculated by its saturation vapor pressure. The equilibrium total moles in the gas phase (n/mol) can be calculated by equation (7)

$$n = n_{SO_2,2} + n_{CO_2,2} + n_{N_2} + n_{\mathrm{DMSO}} \tag{7}$$

So the equilibrium mole fraction of SO_2 in the gas is

$$y_{SO_2} = \frac{n_{SO_2,2}}{n} \tag{8}$$

where y_{SO_2} is the equilibrium SO_2 mole fraction in the gas phase.

$$y_{CO_2} = \frac{n_{CO_2,2}}{n} \tag{9}$$

where y_{CO_2} is the equilibrium CO_2 mole fraction in the gas phase.

The measured pure component constants are given in table 1 [9,10].

The densities of pure solvent DMSO over the temperature range from 293.15 to 343.15 K were determined using a 10 cm^3 gravity bottle with a specially made thermometer. The density was determined by mass using an analytical balance (type TG332A, China). The balance had a range of measurement up to 20 g with an accuracy of ±0.00001 g. The temperature was controlled with ±0.05 K of the test temperature. These measurements were compared with data reported by Pacak [11]. Compared with Pacak [11], the standard deviation of the density was 0.00099. The vapor pressure and density of the pure solvent as a function of temperature are listed in table 3 [12].

Table 1. Critical properties, T_c, P_c, and acentric factors, ω, used in the equation of state.

Component	T_c (K)	P_c (kPa)	ω
SO_2	430.8[a]	7880[a]	0.256[a]
CO_2	304.1[a]	73.8[a]	0.239[a]
N_2	126.2[a]	3390[a]	0.039[a]
DMSO	707[b]	5850[b]	0.414[b]

[a] Ref. [9].
[b] Ref. [10].

2.4. *Test of apparatus*

In order to ensure proper operation of the apparatus, the solubility of SO_2 in water was measured and compared with the values reported in the literature [13]. The experimental measurements agreed with the reported values with a mean relative deviation of 2.22%. The measured values are listed in table 2.

3. Results and discussion

The solubility of SO_2 and CO_2 in DMSO are shown in figure 2 and table 5.

3.1. *The comparison of the experimental measurements and the reported values*

The solubility of SO_2 in DMSO was measured and compared with the values reported in the literature [6,7]. The solubility of CO_2 in DMSO was measured and compared

Table 2. Comparison of experimental data and literature data for SO_2 solubility in water.

T (K)	S (mass%)	S(lit.) (mass%)[a]	$(100(S-S(\text{lit.})))/S$
293.15	10.15	9.61	5.32
298.15	8.61	8.33	3.25
303.15	7.24	7.23	0.14
308.15	6.11	6.10	0.16

[a] Ref. [10].

Table 3. Density, ρ, and vapor pressure P^S, of DMSO.

Expression	Source	SD(ρ)[a]
$\rho/\text{g cm}^{-3} = 1.1195 - 0.001\ (T/\text{K} - 273.15)$	This work	0.00099
$\lg P^S/\text{kPa} = 25.62052 - 3539.32/(T/\text{K}) - 6.00000\ \lg(T/\text{K})$	Literature[b]	

[a] $\text{SD}(\rho) = \sqrt{\sum_{i=1}^{n} (\rho_i^{\text{exp}} - \rho_i^{\text{calc}})^2/(n-1)}$; n = number of data points.
[b] Ref. [12].

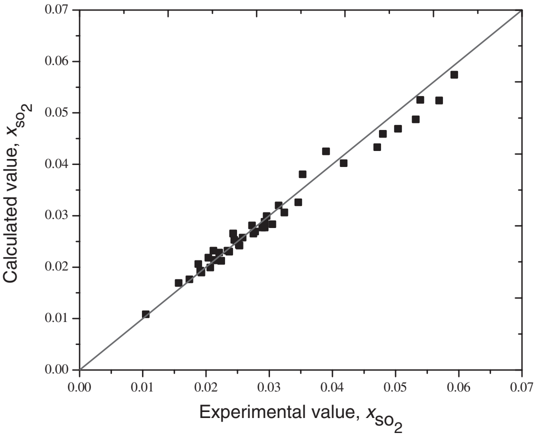

Figure 2. The comparation result of the calculated and experimental values of SO_2 solubility.

Table 4. Comparison of Henry's law constant for SO_2 and CO_2 in polar solvent DMSO at 25°C.

		H (atm)	
Solvent	Solute	Experimental value	Literature value
DMSO	SO_2	0.14	0.109 [6], 0.117 [7]
DMSO	CO_2	109	106 [5]

Note: $H = P_i/x_i$, where P_i is in atm and x_i is the mole fraction of SO_2 and CO_2.

with the values reported in the literature [5]. The comparison of the experimental measurements and the reported values are listed in table 4.

3.2. *Analysis and discussion*

(1) The thermodynamic model:

According to the fundamental thermodynamic relationship that fugacities of a component in coexisting phases are equal at equilibrium, for gas–liquid equilibrium,

$$\hat{f}_i^{\mathrm{g}} = \hat{f}_i^{\mathrm{L}} \tag{10}$$

in the low pressure,

$$\hat{\phi}_i^{\mathrm{g}} y_i p = \gamma_i x_i \phi_i^{\mathrm{s}} p_i^{\mathrm{s}} \tag{11}$$

equation (11) is the basic thermodynamic relationship equation, where p_i^{s} is calculated by the equation of Reid *et al.* [9] (see table 6), x_i, y_i, p are the mole fraction of the liquid, gas and the total pressure. So the key of the model is the calculation of $\hat{\phi}_i^{\mathrm{g}}$, ϕ_i^{s} and γ_i. For the critical properties and acentric factors see table 1.

3.2.1. Fugacity coefficient $\hat{\phi}_i^{\mathrm{s}}$ of pure component i at the system temperature T and p_i^{s}. Fugacity coefficient ϕ_i^{s} may be obtained from equation of the modification of Redlich–Kwong equation of state by Soave for this study [14].

Fugacity coefficient ϕ_i^{s} of pure component following the SRK equation of state is given by:

$$\ln \hat{\phi}_i^{\mathrm{s}} = Z - 1 - \ln(Z - B) - \frac{A}{B}\ln\left(1 + \frac{B}{Z}\right) \tag{12}$$

where:

$$A = \frac{aP}{R^2T^2} \tag{13}$$

$$B = \frac{bP}{RT} \tag{14}$$

$$Z = \frac{PV}{RT} \tag{15}$$

Pure component constants required in equation (12) are given in table 1.

3.2.2. Fugacity coefficient $\hat{\phi}_i^{\mathrm{g}}$ for constituents *i* of a gas mixture.

Fugacity coefficient $\hat{\phi}_i^{\mathrm{g}}$ for constituents i of a gas mixture following the SRK equation of state is given by

$$\ln \hat{\phi}_i^{\mathrm{g}} = \frac{b_i}{b}(Z-1) - \ln(Z-B) - \frac{A}{B}\left[\frac{2\sum_{j=1}^{N} y_j a_{ij}}{a} - \frac{b_i}{b}\right]\ln\left(1+\frac{B}{Z}\right) \tag{16}$$

Table 5. Solubility of SO_2 and CO_2 in DMSO at different temperatures.

T (K)	P (kPa)	y_{SO_2}	y_{CO_2}	y_{DMSO}	x_{SO_2}	x_{CO_2}	$x_{exp}(SO_2)$	$x_{cal}(SO_2)$	$x_{exp}(CO_2)$	$x_{cal}(CO_2)$
293.25	100.88	0.00781	0.180	0.00171	0.0593	0.00167	0.0593	0.0574	0.00167	0.00167
293.15	99.98	0.00711	0.170	0.00172	0.0539	0.00157	0.0539	0.0525	0.00157	0.00160
293.25	100.47	0.00415	0.111	0.00177	0.0315	0.00106	0.0315	0.0320	0.00106	0.00104
293.15	99.28	0.00389	0.0971	0.00178	0.0296	0.000914	0.0296	0.0299	0.000914	0.000890
293.15	100.47	0.00359	0.0781	0.00176	0.0273	0.000746	0.0273	0.0281	0.000746	0.000711
293.25	100.27	0.00323	0.0730	0.00179	0.0245	0.000698	0.0245	0.0253	0.000698	0.000644
293.25	99.49	0.00291	0.0700	0.00180	0.0221	0.000666	0.0221	0.0228	0.000666	0.000593
298.25	100.57	0.00829	0.180	0.00245	0.0569	0.00159	0.0569	0.0524	0.00159	0.00154
298.12	99.70	0.00736	0.171	0.00247	0.0504	0.00151	0.0504	0.0469	0.00151	0.00148
298.15	99.53	0.00497	0.126	0.00252	0.0346	0.00113	0.0346	0.0326	0.00113	0.00109
298.35	99.73	0.00429	0.115	0.00253	0.0305	0.00104	0.0305	0.0283	0.00104	0.000976
298.25	100.29	0.00416	0.111	0.00253	0.0293	0.00101	0.0293	0.0277	0.00101	0.000944
298.05	99.19	0.00397	0.0971	0.00254	0.0275	0.000872	0.0275	0.0265	0.000872	0.000807
298.05	100.26	0.00357	0.0781	0.00254	0.0253	0.000710	0.0253	0.0242	0.000710	0.000641
298.05	100.18	0.00311	0.0730	0.00255	0.0224	0.000666	0.0224	0.0212	0.000666	0.000577
298.15	99.47	0.00293	0.0700	0.00255	0.0207	0.000635	0.0207	0.0199	0.000635	0.000533
303.16	100.45	0.00881	0.180	0.00354	0.0532	0.00154	0.0532	0.0487	0.00154	0.00144
303.16	99.63	0.00781	0.171	0.00367	0.0471	0.00145	0.0471	0.0433	0.00145	0.00137
303.25	99.48	0.00538	0.126	0.00362	0.0324	0.00109	0.0324	0.0306	0.00109	0.000994
303.05	99.73	0.00479	0.115	0.00364	0.0289	0.00100	0.0289	0.0277	0.00100	0.000895
303.10	100.31	0.00461	0.111	0.00364	0.0278	0.000973	0.0278	0.0269	0.000973	0.000862
303.05	99.15	0.00418	0.0971	0.00365	0.0252	0.000842	0.0252	0.0243	0.000842	0.000726
303.15	100.08	0.00392	0.0781	0.00366	0.0237	0.000684	0.0237	0.0230	0.000684	0.000576
303.05	100.03	0.00361	0.0730	0.00367	0.0218	0.000641	0.0218	0.0213	0.000641	0.000525
303.05	99.25	0.00320	0.0700	0.00368	0.0193	0.000611	0.0193	0.0189	0.000611	0.000517
308.15	100.40	0.00945	0.180	0.00495	0.0480	0.00102	0.0480	0.0459	0.00102	0.00112
308.15	99.54	0.00822	0.171	0.00498	0.0418	0.000962	0.0418	0.0402	0.000962	0.00105
308.15	99.43	0.00577	0.126	0.00504	0.0293	0.000720	0.0293	0.0288	0.000720	0.000779
308.15	99.64	0.00509	0.115	0.00506	0.0258	0.000661	0.0258	0.0257	0.000661	0.000712
308.15	100.38	0.00489	0.111	0.00507	0.0248	0.000643	0.0248	0.0250	0.000643	0.000692
308.15	99.27	0.00461	0.0971	0.00508	0.0234	0.000557	0.0234	0.0232	0.000557	0.000598
308.15	100.04	0.00419	0.0781	0.00509	0.0213	0.000452	0.0213	0.0214	0.000452	0.000484
308.15	99.97	0.00376	0.0731	0.00510	0.0191	0.000423	0.0191	0.0193	0.000423	0.000453
308.15	99.22	0.00342	0.0700	0.00511	0.0174	0.000404	0.0174	0.0176	0.000404	0.000431
308.15	99.33	0.00207	0.0560	0.00514	0.0105	0.000325	0.0105	0.0108	0.000325	0.000345
313.20	100.36	0.00985	0.180	0.00692	0.0390	0.000912	0.0390	0.0425	0.000912	0.00112
313.25	99.54	0.00882	0.171	0.00695	0.0353	0.000860	0.0353	0.0380	0.000860	0.00104
313.05	99.51	0.00600	0.126	0.00703	0.0243	0.000643	0.0243	0.0265	0.000643	0.000713
313.05	99.66	0.00520	0.115	0.00705	0.0212	0.000590	0.0212	0.0232	0.000590	0.000621
313.08	100.53	0.00484	0.111	0.00706	0.0204	0.000575	0.0204	0.0218	0.000575	0.000597
313.05	99.50	0.00458	0.0971	0.00707	0.0188	0.000498	0.0188	0.0206	0.000498	0.000549
313.25	100.01	0.00427	0.0781	0.00709	0.0165	0.000377	0.0157	0.0169	0.000377	0.000379

Table 6. The saturated vapor pressure of pure substance.[a]

Substance	Saturated vapor pressure P (bar)	A	B	C	D	Suitable range
SO_2	$\ln P = A - \frac{B}{T} + c \ln T + \frac{DP}{T^2}$	48.882	4552.5	−5.666	990	235K–T_c
CO_2	$\ln \frac{P}{P_c} = (1 - X)^{-1}(AX + BX^{1.5} + CX^3 + DX^6)$ $X = 1 - \frac{T}{T_c}$	−6.95626	1.19695	−3.12614	2.99448	217K–T_c

[a] Ref. [9,12].

For a gas mixture, the following mixing rules apply:

$$a = \sum_i \sum_j y_i y_j a_{ij} \tag{17}$$

$$b = \sum_i y_i b_i \tag{18}$$

where y_i, y_j are the mole fractions of components i and j, a_{ij} is given as:

$$a_{ij} = (a_{ii} a_{jj})^{0.5}(1 - k_{ij}) \tag{19}$$

where k_{ij} is the interaction coefficient given as [16]:

$$k_{ij} = 1 - 2\left(T_{ci} T_{cj}\right)^{1/2} / \left(T_{ci} + T_{cj}\right) \tag{20}$$

3.2.3. Activity coefficient γ_i of component *i*. Activity coefficient γ_i of component i in a liquid solution can be calculated by the NRTL equation [14]. According to the experimental system, and the recommendation of literature [14,15], $\alpha_{12} = 0.3$. For a ternary system, adjustable parameters, τ_{ij}, can be obtained from simplex optimization.

The object function $F = \min \sum_{i=1}^{n} [(\gamma_{\mathrm{cal}} - \gamma_{\mathrm{exp}}/\gamma_{\mathrm{exp}})^2_{SO_2} + (\gamma_{\mathrm{cal}} - \gamma_{\mathrm{exp}}/\gamma_{\mathrm{exp}})^2_{CO_2}]$, and the result of τ_{ij} is listed in table 7.

3.2.4. The test of experimental data. The calculated solubility result with binary interaction coefficient is listed in table 5.

The comparison between the experimental and calculated values is presented in the diagonal chart as shown in figures 2 and 3.

From table 5 and figures 2 and 3, we can see that the solubilities calculated by the model show a good agreement with the experimental data.

4. Conclusions

The solubility of dilute SO_2 and CO_2 in DMSO has been determined in the temperature range from 293.15 to 313.15 K and partial pressure of SO_2 from 0.15 to 2.62 kPa, CO_2 from 5 to 18 kPa using a suitable experimental method and solubility apparatus.

Table 7. Binary interaction coefficient.

Component	τ_{ij}
SO_2–DMSO	1.85
DMSO–SO_2	−4.35
CO_2–DMSO	−3.11
DMSO–CO_2	11.2
SO_2–CO_2	7.56
CO_2–SO_2	2.52

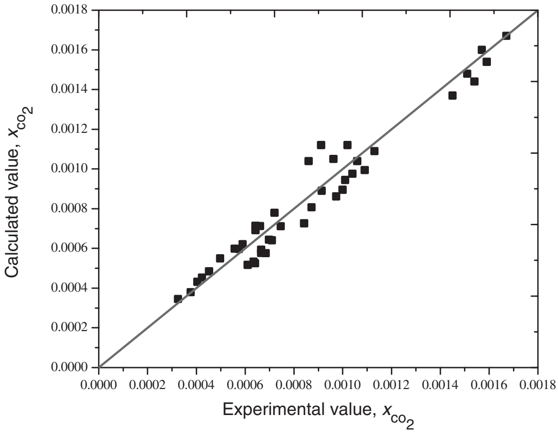

Figure 3. The comparation result of the calculated and experimental values of CO_2 solubility.

The result shows that the solubility of dilute SO_2 in DMSO is high, and that of CO_2 in DMSO is low; the absorption solvent has good selectivity for SO_2 and CO_2.

A solubility model based on gas–liquid phase equilibrium principles is proposed, and the solubilities calculated by the model show good agreement with the experimental data.

The experimental solubility and correlation equation in this article can be used as essential data and model to serve the process design of removal of SO_2 from flue gas.

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