frontiers in Earth Science

BRIEF RESEARCH REPORT
published: 07 October 2021
doi: 10.3389/feart.2021.767015

Experimental Study on the Elemental Sulfur Solubility in Sour Gas Mixtures

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The investigation of elemental sulfur solubility plays critical roles on sour gas reservoir development. In this paper, the solubility of elemental sulfur was directly measured by static method with gas samples from well M1 of a sour gas reservoir in Sichuan Basin. The results show that the solubility of elemental sulfur ranges from 0.001 g/cm^3 to 0.968 g/cm^3 at 40–98.9 MPa and 15–49.8 MPa. The elemental sulfur solubility increases with increasing temperature and pressure, especially when the pressure is greater than 30 MPa. Moreover, the H_2S content in sour gas mixtures is also an important factor affecting elemental sulfur solubility. The elemental sulfur solubility increases with increasing H_2S content of the sour gas mixtures. The experimental data in this paper display a consistent trend with the reported experimental data. Based on the experimental results, the chrastil-type model, Robert's model and Hu's model were investigated and compared. The results show that the chrastil-type model by fitting experimental data in this paper has less error and higher accuracy in calculating elemental sulfur solubility in M gas reservoir. The chrastil-type models proposed in the literature, meanwhile, are only based on the regression of specific gas components and experimental conditions, which lead to a large error in the calculation of elemental sulfur solubility of sour gas samples in this research. The research results provide important basic data and technical support for the development of M gas reservoir.

Keywords: elemental sulfur, solubility, sour gas, H_2S content, chrastil model

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Specialty section:
This article was submitted to Economic Geology, a section of the journal Frontiers in Earth Science

Received: *30 August 2021*
Accepted: *23 September 2021*
Published: *07 October 2021*

Citation:
Zhang R, Gu S, Huang L, Zeng D, Li T and Zhang G (2021) Experimental Study on the Elemental Sulfur Solubility in Sour Gas Mixtures. Front. Earth Sci. 9:767015. doi: 10.3389/feart.2021.767015

INTRODUCTION

Natural gas reservoirs with high sulfur content have huge resource prospects all over the world. Meanwhile, the development of high-sulfur gas reservoirs is also facing great challenges. In these different challenges, elemental sulfur deposition in formation is one of the main problems in gas development process (Kuo, 1972; Chesnoy and Pack, 1997; Roberts, 1997; Roberts, 2017). As the concentration of elemental sulfur in the sour gas exceeds the critical dissolved concentration during the development of sour gas reservoirs, sulfur deposition may occur in the formation. The characteristics of sulfur dissolution, like hydrocarbon dissolution in organic matter (Huang et al., 2020; Huang et al., 2021), are crucial in the analysis of elemental sulfur deposition limit and deposition amount (Smith et al., 1970; Brunner and Woll, 1980; Brunner et al., 1988). Thus, many researchers have been devoted to the study on the elemental sulfur solubility in sour gas from experiment (Kennedy and Wieland, 1960; Roof, 1971; Swift and Manning, 1976; Brunner and Woll, 1980; Brunner et al., 1988; Davis et al., 1993; Gu et al., 1993; Sun and Chen, 2003; Zeng et al., 2005; Yang et al., 2009; Bian et al., 2010; Serin et al., 2010; Cloarec et al., 2012), thermodynamic model (Karan et al., 1998; Heidemann et al., 2001; Cézac et al., 2007; Cézac et al., 2008), semi empirical model (Chrastil, 1982; Eslamimanesh et al., 2011a; Hu et al., 2014; Guo and Wang, 2016), artificial intelligence algorithm (Mohammadi and Richon, 2008; Mehrpooya et al., 2010; Aminian, 2011; Bian et al., 2018; Fu et al., 2019; Bemani et al., 2020; Chen et al., 2020) and molecular simulation (Kadoura et al., 2013; Chen et al., 2019).

Kennedy and Wieland (Kennedy and Wieland, 1960) first measured the elemental sulfur solubility in pure CH_4, CO_2, H_2S and sour gas mixtures. Their results showed that the solubility of sulfur increased with increasing temperature and pressure. The H_2S solution presented the largest elemental sulfur solubility, while the CH_4 solution showed the smallest solubility. However, their experimental method was questioned by roof (Roof, 1971). Subsequently, Roof (Roof, 1971) also measured the elemental sulfur solubility in H_2S, and found that with the increase of temperature, the elemental sulfur solubility initially increased and then presented a decreasing trend as the solubility reached a critical point. However, the sulfur solubility experiment was limited to low temperature and low pressure conditions. Since then, Swift (Swift and Manning, 1976) and Brunner (Brunner and Woll, 1980; Brunner et al., 1988) have reported the measurement of sulfur solubility in pure H_2S under extended temperature and pressure conditions. Davis (Davis et al., 1993) extended the dissolution of sulfur in typical components to complex multi-component sour gas, and broadened the temperature and pressure range of the experiment. Gu (Gu et al., 1993) used a set of static method equipment to determine the solubility data of sulfur in CH_4, CO_2, H_2S and their mixtures. Sun (Sun and Chen, 2003), Zeng (Zeng et al., 2005), Yang (Yang et al., 2009) and Bian (Bian et al., 2010) also expanded the range of the solutions and the limits of experimental conditions. Serin (Serin et al., 2010) measured the elemental sulfur solubility in pure CO_2 under lower temperature and pressure conditions. Cloarec (Cloarec et al., 2012) measured the elemental sulfur solubility in pure CH_4 by improving the capture device and gas expansion device, and their results were slightly different from those of Kennedy and Wieland.

The published experiments on the elemental sulfur solubility are summarized in **Appendix A**. With the advancement of experimental methods, the experimental pressure and temperature ranges have been extended to 0.5–138 MPa and 303.2–563 K, and the solutions have been extended from single-component H_2S, CO_2, CH_4 to multi-component wellhead gas sample of the gas fields. These experimental results provide crucial data for the solubility model. However, due to the huge experimental costs, as well as the potential experimental risks from toxic gas, the experimental data of elemental sulfur solubility are far from sufficient. Especially, the experimental data of elemental sulfur solubility in sour gas mixtures from high-containing sulfur gas well in China remain limited. Also, owing to the small solubility value, the sulfur solubility is susceptible to various interferences, such as temperature, pressure, and H_2S content (Eslamimanesh et al., 2011b; Luo et al., 2012; Eslamimanesh et al., 2013). Some documented experimental work has reported inconsistent results, the elemental sulfur solubility urgently needs further verification and supplementation. The theoretic model of sulfur solubility can overcome the experimental demerits like huge cost, long period, and high risk, and thus predict the sulfur solubility conveniently. Nevertheless, the accuracy of reported theoretic models of sulfur solubility in sour gas samples from Chinese high-sulfur gas wells remains to be verified. Accordingly, this wok is aimed to supplement key sulfur solubility data in sour gas samples from Chinese high-sulfur gas well. The effects of temperature, pressure, and H_2S content are analyzed. Combined with the measured data, the accuracy of the common chrastil-type model and its extended models are further explored.

EXPERIMENTAL METHODOLOGY

Principles and Apparatus

Based on the principle of solvent dissolution, the sour gas sample is pressed into the sample reaction vessel by the piston pump, the sample reaction vessel can simulate the high temperature and high pressure of the formation. Subsequently, fully reacted and mixed sour gas sample is passed through the CS_2 solution, CS_2 solvent is a commonly used elemental sulfur solvent, the elemental sulfur in the sour gas sample is dissolved in CS_2 solution and the content of elemental sulfur in the CS_2 solution is measured with a gas chromatograph to calculate the sulfur content in the original gas sample. After that, the H_2S in the mixed gas is absorbed by the NaOH solution from a security perspective, and the gas flow meter can measure the volume of gas mixtures without H_2S at room temperature and pressure. The experimental apparatus is shown in **Figure 1**. The key parts of the apparatus are: 1) gas mixing system; 2) high temperature and high pressure resistant experimental reaction vessel; 3) gas absorption device; 4) gas chromatograph. Finally, the sulfur solubility of gas mixtures can be calculated by the following formula:

$$c = \frac{mS}{100V} \tag{1}$$

Where c is sulfur solubility, g/m^3; m is CS_2 solvent quality, g; S is elemental sulfur content in CS_2 solvent, %; V is volume of gas mixtures, m^3.

Gas Sample and Determination of Elemental Sulfur Content

The gas sample is taken from a downhole gas sample from well M1 in a sour gas field in the Sichuan Basin. The sampling depth is 3800 m, the sampling pressure is 35 MPa, and the temperature is 98.1°C. The temperature and pressure are restored indoors, and the opening pressure is checked. The H_2S content is determined by titration for three times. The molar contents of the three titrations are 17.982%, 17.891 % and 17.668%. Take the middle value of three titration, the H_2S content of the downhole sample

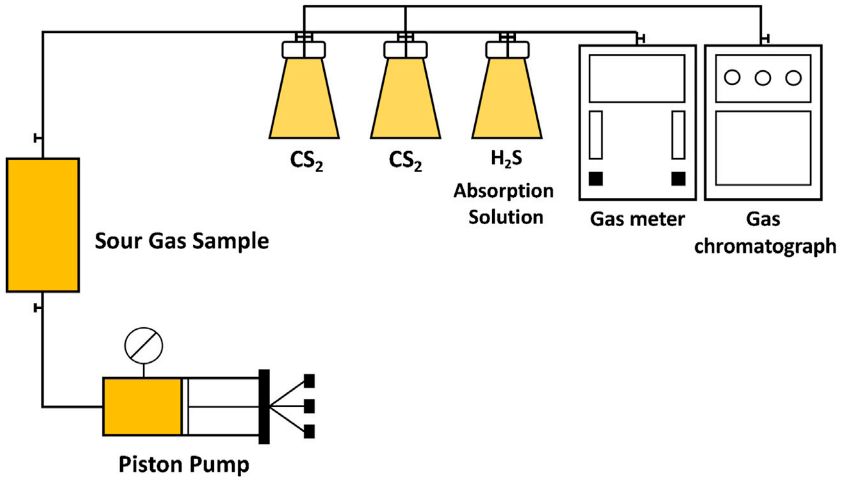

FIGURE 1 | The experimental apparatus for measuring elemental sulfur content

TABLE 1 | Solubility of elemental sulfur in well M1 by experiments and predictions.

Pressure (MPa)	Elemental sulfur solubility (g/m^3)						
	Measured data				Predicted data		
	98.9°C	80°C	60°C	40°C	Hu's model[a]	Roberts's model[b]	This paper[c]
49.8	0.968	0.429	0.175	0.076	0.0012	369.7	0.829
40	0.550	0.225	0.095	0.040	0.00083	227.4	0.523
30	0.220	0.104	0.039	0.015	0.00043	101.7	0.243
15	0.031	0.008	0.002	0.001	0.00017	7.2	0.019

[a] *Model coefficients (see* ***Eq. 2****): k = 1.592, a = −2737, b = −8.89768 (ρ < 200); k = 3.288, a = −4880, b = −12.4969 (ρ > 200).*
[b] *Model coefficients: k = 4, a = −4666, b = −4.5711.*
[c] *Model coefficients: k = 3.8, a = −6648, b = −4.7293.*

of well M1 is 17.891%, which is a high-sulfur content gas reservoir. The composition of the natural gas in well M1 can be obtained by gas chromatograph. The CH_4 content in the gas sample is 73.418%, the CO_2 content is 8.66%, the C_2H_6 content is 0.032%, and the contents of other components are negligible.

To determine the sulfur content of a gas sample, a group of solutions with known sulfur content are used for calibration. In order to cover the measured range of elemental sulfur solubility, high-concentration standard samples were prepared. The sulfur contents are 0.005%, 0.01%, 0.03% and 0.05% respectively. The standard peak areas of different sulfur contents are used as the reference values. Then the differences between the measured peak area and the standard peak areas are used to obtain the sulfur content of the gas sample.

The elemental sulfur contents of the gas in well M1 are determined by experiments. The original elemental sulfur content of the gas sample in downhole under formation condition is 0.091 g/m^3, the elemental sulfur content under sampling conditions (35 MPa, 98.1°C) is 0.357 g/m^3, and the saturated sulfur content under formation conditions is 0.968 g/m^3. Thus, the elemental sulfur in the gas sample has not reached saturation under the formation conditions and the sampling conditions.

RESULTS AND DISCUSSION

Solubility of Elemental Sulfur in Well M1

The elemental sulfur solubility experiment was carried out using gas samples from well M1, the solubility of elemental sulfur was measured among 40–98.9°C and 15–49.8 MPa. The elemental sulfur solubility was determined by taking the average of three tests for each pressure point. The errors among different measurements were checked to be smaller than 2%. The experimental results are shown in **Table 1** and **Figure 2A**. It can be seen from **Figure 2A** and **Table 1** that at the same temperature, the solubility of sulfur increases with increasing pressure; and at the same pressure, the solubility of elemental sulfur increases as the temperature increases. At the formation temperature (98.9°C), the solubility of elemental sulfur reaches 0.968 g/m^3 at 49.8 MPa and then drops to 0.031 g/m^3 as the pressure decreases to 15 MPa. At the

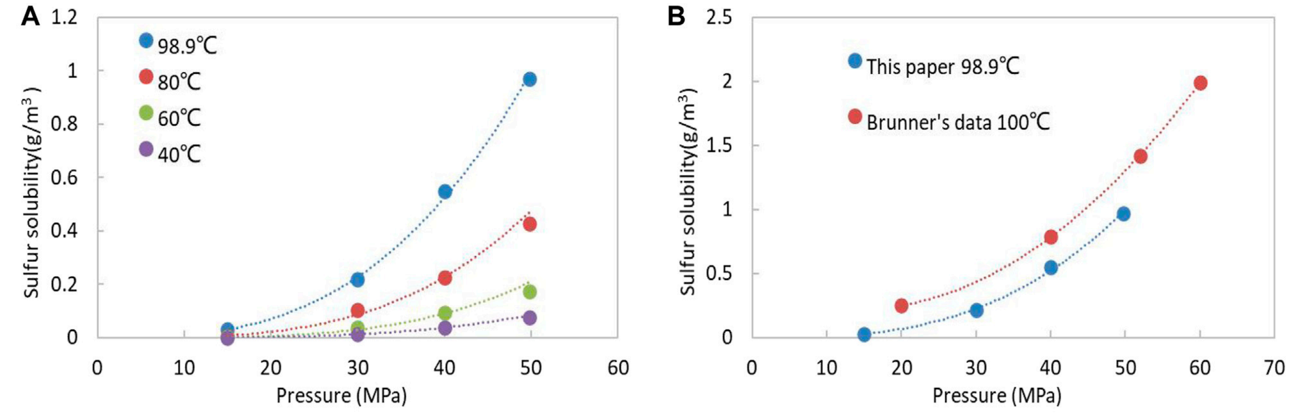

FIGURE 2 | Sulfur solubility in well M1 gas sample. **(A)** experimental results in this work; **(B)** comparison with Brunner's data at similar gas compositions. The sour gas in Brunner's data contains 20 % H_2S, 66 % CH_4, 10 % CO_2, and 4 % N_2.

formation pressure (49.8 MPa), the solubility of elemental sulfur drops to 0.076 g/m^3 with the temperature decreasing to 40°C. The elemental sulfur solubility is sensitive to variations of temperature and pressure. The experimental results of sulfur solubility at 98.9°C are compared with Brunner's data at 100°C in **Figure 2B** (Brunner and Woll, 1980). The sour gas mixtures in Brunner's data possess similar gas compositions (20% H_2S, 66% CH_4) with the gas samples from well M1. As seen from **Figure 2B**, the experimental data in this paper are basically consistent with the Brunner's data. The sulfur solubility in the literature is slightly higher than the measured results in this work, the deviations can be well attributed to the slightly higher H_2S content and temperature in Brunner's gas sample than that in this paper.

Chrastil Model Fitting

Chrastil derived solubility correlation of solids and liquids in supercritical gases base on the law of mass action (Chrastil, 1982). Chrastil model was used to study the solubility of various food additives in supercritical CO_2 fluids in the early days. Subsequently, Roberts (Roberts, 1997) used the Chrastil solubility prediction model for the first time to study the solubility of elemental sulfur in sour gases containing H_2S. By fitting the experimental data in the literature, the Chrastil prediction model for the solubility of elemental sulfur in sour gases was obtained. Since then, the Chrastil solubility prediction model has been widely used in the fitting of existing sulfur solubility experimental data, and the elemental sulfur solubility prediction model suitable for different temperatures and pressures and different gas components has been obtained (Eslamimanesh et al., 2011a; Hu et al., 2014; Guo and Wang, 2016). The expressions of this correlation are as follows, the detailed derivation process of this correlation and coefficients can be found in the Chrastil's paper (Chrastil, 1982):

$$c = \rho^k e^{(a/T+b)} \tag{2}$$

$$a = \frac{\Delta H}{R} \tag{3}$$

$$b = \ln(M_A + kM_B) + q - k\ln M_B \tag{4}$$

Where ρ is gas density, kg/m^3; T is temperature, K; k is an association number, which denotes a solute A associates with k molecules of a gas B to form one molecule of a solvato complex AB_k in equilibrium with the system (Chrastil, 1982); a and b are constant coefficients; ΔH is the total reaction heat, kJ/mol; R is gas constant, 8.314 J·mol^{-1}·K^{-1}; M_A、 M_B are the molecular weights of the solute and of the gas, correspondingly; q is a constant. Subsequently, Roberts obtained the constant coefficients of the expression by fitting experimental data (Roberts, 1997):

$$c = \rho^4 e^{(-4666/T-4.5711)} \tag{5}$$

However, Roberts's model has a limited applicability due to the temperature and pressure range and gas composition. Consequently, Hu proposed a segmentation fitting method according to different gas density ranges by using published data (Hu et al., 2014). Meanwhile, the three coefficients in **Eq. 1** are obtained according to the above fitting method using this experiment data, The fitting process and method are detailed in Hu's article (Hu et al., 2014), the results are compared and shown in **Figure 3A,B** and **Table 1**. As seen from **Figure 3A,B** and **Table 1**, the chrastil-type model fitted in this paper has a comparatively well prediction effect, while the predicted results of the Roberts's and Hu's models deviate seriously from the experiment data, which indicates the values of coefficients a and b have a great influence on the predicted solubility results due to the temperature and pressure conditions and different components of the sour gas mixtures.

The association number k will change with the system temperature and pressure. As the temperature increases at a constant pressure, the molecular thermal motion becomes more violent, and the association number k decreases; when the temperature is a constant and the pressure increases, the molecular distance is shortened, the chance of intermolecular

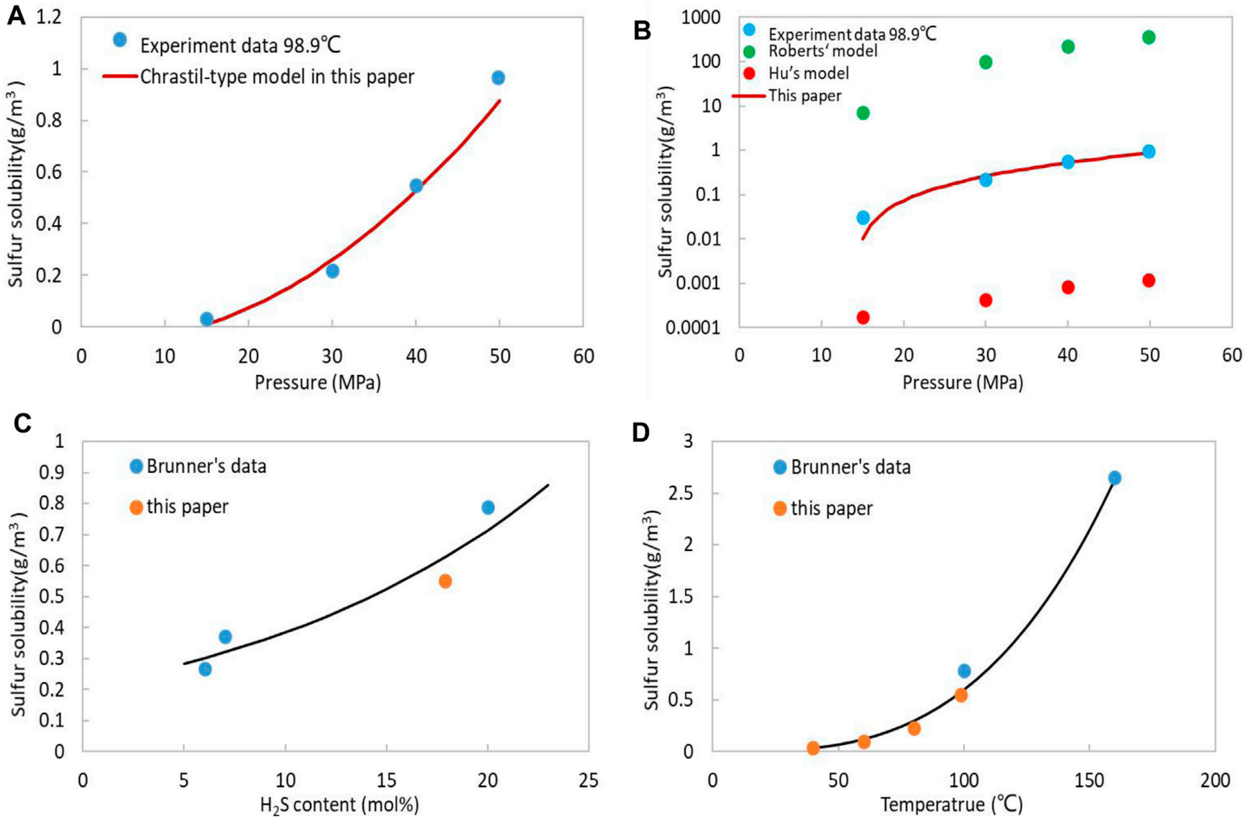

FIGURE 3 | Comparison of sulfur solubility with published data. **(A)** Effect of pressure at 98.9°C; **(B)** Experimental sulfur solubility of this paper and published data. **(C)** Effect of H_2S content at 40 MPa, 100°C; **(D)** Effect of temperature at 40 MPa.

collisions increases, and the association number k increases. ΔH will change accordingly with the association number k. Therefore, the practice of treating k, a, and b as constants will inevitably lead to larger deviations. In order to improve the accuracy of the model, it is necessary to obtain the values of k, a, and b within a specific range of temperature, pressure, and gas composition.

H_2S Content and Temperature

In order to investigate the influence of H_2S content on the solubility of elemental sulfur, the sulfur solubility data at 40 MPa and 100°C in the published literature (Brunner and Woll, 1980) were compared with the measured results of sour gas mixtures from M1 well at 40 MPa and 98.9°C, as shown in **Figure 3B**. The results show that under the same pressure and temperature conditions, as the molar content of H_2S increases, the dissolved elemental sulfur content in the sour gas mixtures increases. The experimental results in this paper are in good agreement with the published experimental data.

Similarly, the influence of temperature on the solubility of elemental sulfur is also investigated. The sulfur solubility data of the sour gas mixtures at 40 MPa and 100–160°C in the published literature (Brunner and Woll, 1980) were compared with the measured results of sour gas mixtures from M1 well at 40 MPa and 40–98.9°C, as shown in **Figure 3C**. The H_2S molar content of the sour gas mixtures in Brunner's work is 20%, which is close to the H_2S content of the sour gas mixtures in this work. The results show that under the same pressure, as the temperature increases, the dissolved elemental sulfur content in the sour gas mixtures increases. The experimental results also show good consistency with Brunner's data.

CONCLUSION

In this study, the elemental sulfur solubility was measured by the static method within sour gas mixtures samples from M1 well in Sichuan Basin. The results show that the experimental data in this work show good agreement with the published results. The sulfur solubility increases with increasing pressure and temperature. At the same pressure and temperature conditions, the sulfur solubility increases as the H_2S molar content increases. The reported chrastil-type model with coefficients fitted by the measured results can well predict the sulfur solubility of gas sample from M1 well, while the presented chrastil-type formulas with documented coefficients fail to obtain reasonable prediction results. Owing to the great influence of temperature on sulfur solubility, wellbore electric heating can be utilized to prevent sulfur deposition and plugging in the wellbore, and improve sulfur recovery in the middle-late stage of sour gas reservoir development.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author.

AUTHOR CONTRIBUTIONS

RZ: Data creation, Formal analysis, Writing-original draft, Writing-review and editing, Funding acquisition. SG: Data creation, Visualization, Writing-review and editing. LH: Conceptualization, Project administration, Supervision, Writing-review and editing. DZ: Conceptualization, Funding acquisition, Project administration. TL: Investigation, Methodology, Writing-original draft. GZ: Formal analysis, Validation.

ACKNOWLEDGMENTS

The financial supports of National Natural Science Foundation of China (U19B6003) are acknowledged. The helpful comments and suggestions by the reviewers are greatly appreciated.

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Conflict of Interest: Author RZ, SG, DZ, and TL were employed by SINOPEC. The remaining author declares that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

APPENDIX A THE EXPERIMENTAL SUMMARY FOR DETERMINATION OF SULFUR SOLUBILITY.

Researcher	Time	Temperature (K)	Pressure (MPa)	Gas components
Kennedy and Wieland, (1960)	1960	338.71–394.26	6.89–41.35	Pure CH_4, CO_2, H_2S and sour gas mixtures with different proportions
Roof, (1971)	1971	316.46–383.15	7–31.15	H_2S
Swift and Manning, (1976)	1976	394–450	34.5–138	H_2S
Brunner and Woll, (1980)	1980	373.15–433.15	10–60	H_2S and H_2S-CO_2-CH_4-N_2 mixtures
Brunner et al. (1988)	1988	398–486	6.7–155	H_2S-CO_2-CH_4-N_2-C_2H_6-C_4H_{10} mixtures
Davis et al. (1993)	1992	333–425	5–55	Sour gas mixtures with different proportions
Gu et al. (1993)	1993	353.2–383.3	20.52–50.17	Pure CH_4、CO_2、H_2S and mixtures
Sun and Chen, (2003)	2003	303.2–363.2	20–45	Pure CH_4、CO_2、H_2S and mixtures
Zeng et al. (2005)	2005	353.15–433.15	10–60	H_2S-CO_2-CH_4-N_2-C_2H_6-C_4H_{10}-C_6H_{14} mixtures
Yang et al. (2009)	2009	373.15	16–36	Wellhead gas sample of a gas well (mixtures)
Bian et al. (2010)	2010	336.2–396.6	10–55.2	Wellhead gas sample of a gas well (mixtures)
Serin et al. (2010)	2010	333.15/393.15	0.93–29.45	CO_2
Cloarec et al. (2012)	2012	363.15	4–25	CH_4