Effect of H₂S on the Corrosion Behavior of Pipeline Steels in Supercritical and Liquid CO₂ Environments

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ABSTRACT

The objective of the present study is to evaluate the corrosion properties of pipeline steels in $CO_2/H_2S/H_2O$ mixtures with different amounts of water (undersaturated and saturated) related to a natural gas transportation pipeline. Corrosion behavior of carbon steel, 1Cr steel, and 3Cr steel was evaluated using an autoclave with different combinations of CO₂ partial pressure and temperature (8 MPa/25°C and 12 MPa/80°C) with 200 ppm H_2S . The corrosion rate of samples was determined by weight-loss measurements. The surface morphology and the composition of the corrosion product layers were analyzed using surface analytical techniques (scanning electron microscopy and energy dispersive x-ray spectroscopy). Results showed that the corrosion rate of materials in supercritical and liquid phase CO₂ saturated with water was very low (<0.01 mm/y). However, adding 200 ppm of H_2S to the supercritical and liquid CO2 system caused mild corrosion (<0.5 mm/y). Reducing water content to 100 ppm in the supercritical and liquid CO_2 systems with 200 ppm of H_2S reduced the corrosion rate to less than 0.01 mm/y.

KEY WORDS: carbon steel, CO $_2$ corrosion, H $_2$ S, low Cr steel, supercritical/liquid CO $_2$

INTRODUCTION

Development of natural gas fields containing large quantities of CO_2 must consider the separation of CO_2 from the natural gas so that it complies with the technical standards necessary to provide the natural gas to market.¹ Usually, conventional CO_2 separation technologies remove CO_2 from natural gas at low pressure and release it to the atmosphere.² However, as a result of the large quantities of CO_2 present in the high-pressure CO_2 gas fields, the CO_2 must be captured and transported to sequestration sites separately, which presents similar challenges as seen in CO_2 transmission related to carbon capture and storage.

It has been acknowledged that dry supercritical and liquid CO₂ are not corrosive. However, corrosion rates are much higher if free water is present because of its reaction with CO₂ to form a high concentration of corrosive species. As a result of the direct impact of the presence of formation water and high-pressure CO_2 on the corrosion of pipeline steel, studies related to aqueous CO₂ corrosion at high CO₂ pressure have recently been conducted. It has been reported that the corrosion rate of carbon steel under high CO_2 pressure (liquid and supercritical CO₂) without formation of protective FeCO3 corrosion product layers is very high $(\geq 20 \text{ mm/y})$.³⁻⁷ At certain conditions, the corrosion rate can decrease to low values (<1 mm/y) after long-term exposures as a result of the formation of a protective layer of $FeCO_3$.⁸⁻¹⁰ In addition, recent studies have reported that the presence of trace impurities, such as SO_x and NO_x, can cause significant corrosion for carbon steel in supercritical and liquid

Submitted for publication: January 11, 2016. Revised and accepted: March 30, 2016. Preprint available online: March 30, 2016, http:// dx.doi.org/10.5006/2026.

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FIGURE 1. Schematic of different parts of the pipeline with inlet and outlet conditions.

 $\rm CO_2$ in the presence of small amounts of H₂O (below the solubility limit).¹¹⁻¹⁸ Related to gas field development, it has recently been reported that there can be small amounts of H₂S present in the high-pressure CO₂ streams, whereas the effect on corrosion has thus far not been studied.

Figure 1 shows a schematic of a CO_2 transportation pipeline experiencing a temperature drop. At the inlet condition, the pressure is 12 MPa and temperature is 80°C. At this condition, CO_2 is in the supercritical phase. Along the pipeline the temperature drops and, consequently, pressure drops and the CO_2 transitions from supercritical to liquid phase. Supercritical CO_2 at a pressure of 12 MPa and temperature of 80°C can dissolve 10,000 ppm of water.¹⁹ However, liquid CO_2 at 8 MPa and 25°C can dissolve only 3,000 ppm water.¹⁹ Therefore, temperature drop and, consequently, CO_2 phase transformation cause the formation of free water in the system.

Thus, the objective of the present study was to evaluate the corrosion performance of pipeline steels in supercritical and liquid CO_2 phases with and without temperature fluctuations and water condensation and also with and without H₂S. Corrosion behavior of carbon steel, 1Cr steel, and 3Cr steel was evaluated using an autoclave with different combinations of CO_2 partial pressure and temperature (8 MPa/25°C and 12 MPa/80°C) at a 200 ppm H₂S concentration.

EXPERIMENTAL PROCEDURES

The materials used in this work are as follows:

- UNS K03014⁽¹⁾ carbon steel, named CS;
- UNS G41300 1Cr steel, named 1Cr; and
- UNS G41300 3Cr steel, named 3Cr.

All materials were analyzed for chemical composition using atomic emission spectroscopy. Table 1 shows chemical compositions of the three materials used in the present study.

Chemical Compositions of Materials Used in the Present Study (wt%, balance Fe)

TABLE 1

	С	Cr	Mn	Р	S	Si	Cu	Ni	Мо	AI
CS	0.065	0.05	1.54	0.013	0.001	0.25	0.04	0.04	0.007	0.041
1Cr	0.3	0.85	0.91	0.015	0.008	0.29	_	—	_	—
3Cr	0.08	3.43	0.54	0.006	0.003	0.3	0.16	0.06	0.32	—



FIGURE 2. Schematic of specimen location in the autoclave.

The specimens for the corrosion tests were machined to be rectangular shape with a size of $1.27 \text{ cm} \times 1.27 \text{ cm} \times 0.254 \text{ cm}$. A 5 mm diameter hole at one end served to hang the samples from a sample stand with a nonmetallic washer. The specimens were ground with 600 grit silicon carbide (SiC) paper, cleaned with isopropyl alcohol (i-C₃H₇OH) in an ultrasonic bath, dried, and weighed using a balance with a precision of 0.1 mg.

The corrosion experiments were performed in a 7.5 L autoclave (UNS N10276). The electrolyte was a 1 wt% NaCl solution. In the present study, the corrosion behavior of materials was evaluated in CO_2 -rich phase (Figure 2), where samples were located in the CO_2 phase. Water content at the bottom of the autoclave was varied in correspondence with the water concentration in the CO_2 phase. Once sealed, the autoclave temperature was adjusted. Then, a mixture of CO_2 and H_2S was directly injected into the autoclave to the desired H_2S concentration (200 ppm). Finally, high-pressure CO_2 was added to the autoclave with a gas booster pump to the desired working pressure.

The corrosion rates were determined from the weight-loss method at the end of the test. In each test, two specimens were simultaneously exposed to the corrosive environment in order to obtain an average corrosion rate. The specimens were removed and cleaned for 5 min in Clarke's solution (20 g antimony trioxide + 50 g stannous chloride and hydrochloric acid to make 1,000 mL). The specimens were then rinsed in distilled water, dried, and weighed to 0.1 mg. The average corrosion rate during the test period can be calculated by the following equation:²⁰

⁽¹⁾ UNS numbers are listed in *Metals and Alloys in the Unified Numbering System*, published by the Society of Automotive Engineers (SAE International) and cosponsored by ASTM International.

	Material	pCO ₂ (MPa)	H₂S (ppm)	Temperature (°C)	Duration (h)	Water Content ^(A)
H ₂ O saturated	CS	12	0	25	24	saturated
	CS	12	0	80	24	saturated
	1Cr	12	0	25	24	saturated
	1Cr	12	0	80	24	saturated
H ₂ S & H ₂ O saturated	CS	12	200	80	48	saturated
	1Cr	12	200	80	48	saturated
	3Cr	12	200	80	48	saturated
	CS	8	200	25	48	saturated
	1Cr	8	200	25	48	saturated
	3Cr	8	200	25	48	saturated
H ₂ S & H ₂ O	CS	12	200	25	24	100 ppm
undersaturated	CS	12	200	80	24	100 ppm
	1Cr	12	200	25	24	100 ppm
	1Cr	12	200	80	24	100 ppm

 TABLE 2

 Test Conditions for Corrosion Study in the CO₂-Rich Phase Without Condensation

^(A) $ppm = ppm_v$.



FIGURE 3. Experimental procedures for evaluating the dewing corrosion behavior of materials in high pCO_2 environments with H_2S .

Corrosion rate (mm/y)

$$= \frac{8.76 \times 10^4 \text{ (mm} \cdot \text{h/cm} \cdot \text{y}) \times \text{weight loss (g)}}{\text{area (cm^2)} \times \text{density (g/cm^3)} \times \text{time (h)}}$$
(1)

The morphology and compositions of corrosion products were analyzed using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS).

Corrosion in CO₂ Phase Without Water Condensation

Table 2 shows the test matrix for corrosion of materials in supercritical/liquid CO_2 phases with different amounts of water and H_2S . For the water-saturated CO_2 condition, 10 g of 1 wt% NaCl solution was added to the autoclave in order to ensure saturation. Tests with 100 ppm of water represent undersaturated conditions.

Corrosion in CO₂ Phase with Water Condensation

Figure 3 shows experimental procedures for evaluating dewing corrosion behavior of materials at high pCO_2 conditions. Initially, 10 g of 1 wt% NaCl solution were added to the autoclave in order to ensure saturation. The decrease in temperature during experiments will cause water condensation on the

 TABLE 3

 Test Conditions for Dewing Corrosion Study

	Material	pCO ₂ (MPa)	H ₂ S (ppm) ^(A)	Temperature (°C)	Water Content
Dewing	CS	12	0	$80 \rightarrow 25$	saturated
•	1Cr	12	0	$80 \rightarrow 25$	saturated
	3Cr	12	0	$80 \rightarrow 25$	saturated
	CS	12	200	$80 \rightarrow 25$	saturated
	1Cr	12	200	$80 \rightarrow 25$	saturated
	3Cr	12	200	$80 \rightarrow 25$	saturated

^(A) $ppm = ppm_v$.

specimen surface and provide a condition for dissolving CO_2 and H_2S therein. It is important to note that only one cycle of dewing was simulated experimentally. Table 3 shows the test matrix for the dewing corrosion study.

RESULTS AND DISCUSSION

Corrosion in CO₂ Phase Without Water Condensation

Table 4 shows the summary of corrosion rate data of three different steels in the supercritical CO_2 phase (inlet condition) and liquid CO_2 phase (outlet

	Material	pCO ₂ (MPa)	H ₂ S (ppm)	Temperature (°C)	Water Content	Corrosion Rate (mm/y) ^(A)
H ₂ O saturated	CS	12	_	25	saturated	<0.01
	CS	12	_	80	saturated	<0.01
	1Cr	12	_	25	saturated	<0.01
	1Cr	12	_	80	saturated	<0.01
H ₂ S & H ₂ O saturated	CS	12	200	80	saturated	0.41
	1Cr	12	200	80	saturated	0.44
	3Cr	12	200	80	saturated	0.05
	CS	8	200	25	saturated	0.07
	1Cr	8	200	25	saturated	0.13
	3Cr	8	200	25	saturated	0.14
H ₂ S & H ₂ O	CS	12	200	25	100 ppm	<0.01
undersaturated	CS	12	200	80	100 ppm	<0.01
	1Cr	12	200	25	100 ppm	<0.01
	1Cr	12	200	80	100 ppm	<0.01

 TABLE 4

 Summary of Corrosion Rate Data of Three Different Steels in CO₂ Phase

^(A) Standard deviation of corrosion rate was within 5%.



FIGURE 4. SEM image and EDS spectra of the surface of CS after exposure to water-saturated supercritical CO_2 (12 MPa, 80°C) with 200 ppm of H_2S .

condition) with and without H₂S. Experimental data show that corrosion rate in supercritical and liquid CO₂ phases saturated with water is very low (<0.01 mm/y), consistent with previous results.^{11-12,21-22} However, adding H₂S to the supercritical and liquid CO₂ systems leads to corrosion. Furthermore, reducing water content to 100 ppm in supercritical and liquid CO₂ systems with 200 ppm H₂S reduced the corrosion rate to less than 0.01 mm/y.



FIGURE 5. Result of XRD analysis for CS after exposure to watersaturated supercritical CO₂ (12 MPa, 80°C) with 200 ppm of H_2S .

Figure 4 shows SEM image and EDS spectra of the corroded CS sample surface, exposed to watersaturated supercritical CO_2 (12 MPa, 80°C) with 200 ppm of H₂S. It can be seen that the surface was covered by corrosion products that consisted of Fe and S. This indicates the formation of FeS on the steel surface under this condition, confirmed by x-ray diffraction (XRD) analysis shown in Figure 5. However, XRD analysis also detected iron carbonate (FeCO₃) on the CS sample surface, which was not observed from the surface EDS analysis.

Figure 6 represents a cross-sectional SEM image and EDS spectra of the CS sample exposed to watersaturated supercritical CO_2 (12 MPa, 80°C) with 200 ppm of H₂S. As expected, it shows a bilayer structure: a thin outer layer consisting of Fe and S,



FIGURE 6. SEM image and EDS spectra of the cross section of CS after exposure to water-saturated supercritical CO₂ (12 MPa, 80°C) with 200 ppm of H_2 S.



FIGURE 7. SEM image and EDS spectra of the surface of 3Cr steel after exposure to water-saturated supercritical CO_2 (12 MPa, 80°C) with 200 ppm of H_2S .

and a thick/continuous inner layer consisting of Fe, C, and O. Combined with the result of XRD analysis (Figure 5), the outer layer can be identified as mackinawite (FeS) and the inner layer as FeCO₃. A similar morphology was observed for 1Cr steel. Figure 7 shows the SEM image and EDS spectra of the corroded surface of 3Cr steel after exposure to water-saturated supercritical CO_2 (12 MPa, 80°C) with 200 ppm of H₂S. It can be seen that the surface was covered by a thin layer of sulfur-containing corrosion products. Note that the polishing marks are still



FIGURE 8. SEM and EDS analyses of three different steels after corrosion experiments in liquid CO₂ phase saturated with H_2O at 8 MPa, 25°C, containing 200 ppm of H_2S .

visible, indicating that the corrosion of this material was minimal, compare to CS and 1Cr steel.

Figure 8 shows SEM images and EDS line scanning results of the corroded samples (CS, 1Cr, and 3Cr steels) for their surfaces and in cross section after exposure to water-saturated supercritical CO_2 (8 MPa, 25°C) with 200 ppm of H₂S. SEM surface analysis shows a similar morphology for corrosion products for all three steels. EDS elemental analysis shows that the corrosion product layer is mostly FeS. EDS line scanning also shows that for 3Cr steel there is a chromium-rich layer close to the metal surface and underneath the FeS layer. This chromium-rich layer reduces the adherence of corrosion products to the metal surface and, consequently, reduces the protectiveness of the corrosion product layer and increases the corrosion rate. It is interesting to note that under this low-temperature condition (25° C), FeCO₃ was not observed on the surface of the CS and 1Cr steel.

Figure 9 shows the surface appearance of the CS and 1Cr steel samples exposed to the supercritical CO_2 phase (12 MPa, 80°C) with 100 ppm of water and





FIGURE 9. SEM images of the sample surface exposed to the supercritical CO_2 phase (12 MPa, 80°C) with 100 ppm of water and 200 ppm of H_2S for 24 h: (a) CS and (b) 1Cr steel.

 $200 \text{ ppm of } H_2S \text{ for } 24 \text{ h. No visible signs of corrosion}$ were observed on samples, i.e., the surfaces appeared shiny and devoid of any type of corrosion products.

As shown in these results, adding 200 ppm of H_2S increased the corrosion rate of materials in the watersaturated CO₂ phase. This indicates that H_2S can change the adsorbability of water on the steel surface, resulting in formation of a thin water layer on the steel surface.²³ A lower surface energy condition on the steel can result in development of nucleation sites for the formation of water droplets saturated with H₂S and CO₂, which in turn leads to formation of both FeCO₃ and FeS on the steel surface at high temperature (80°C). Increasing the temperature from 25°C to 80°C increased the corrosion rate almost five times because of the increased water content in the CO₂ phase (i.e., from 3,000 ppm to 10,000 ppm of water in the CO₂ phase)¹⁹ and also accelerated the rate of chemical and electrochemical reactions.

Corrosion in CO₂ Phase with Water Condensation (Dewing Corrosion)

Temperature fluctuations in CO_2 transportation pipelines cause phase transitions of CO_2 and consequent water condensation (Figure 1). A summary of corrosion experimental data under these conditions is shown in Table 5. Without any condensation and dewing in the system, no corrosion occurs without H₂S (Table 4). However, under dewing conditions, corrosion occurs both in pure CO_2 and CO_2/H_2S systems, indicating the water condenses on the steel surface.

Figure 10 shows the surface appearance of the CS, 1Cr steel, and 3Cr steel samples exposed to dewing condition without H_2S . For all three materials, the polishing marks were still visible on most of the surface, with some scattered corrosion products. It can be speculated that the corrosion products were formed where a condensed water droplet attached to the steel surface. EDS elemental analysis confirmed that the scattered corrosion products were identified as FeCO₃ in this condition (Figure 11).

The surface morphologies of samples exposed to dewing condition with 200 ppm of H₂S are represented in Figure 12. Comparing with the case without H₂S, it is clearly shown that most of the steel surface was covered by corrosion product when H₂S was present, indicating that condensed water formed a thin and uniform water layer on the steel surface. For all three steels, the corrosion product was identified as FeS by EDS analysis (Figure 13). However, the crosssectional SEM and EDS analysis showed a bilayer structure of the corrosion product: an outer layer consisting of Fe and S, and an inner layer consisting of mainly Fe, C, and O (Figure 14). This indicates the formation of both FeS and FeCO₃ on the steel surface

		, 				
	Material	Initial pCO ₂ (MPa)	H ₂ S (ppm)	Temperature (°C)	Water Content	Corrosion Rate (mm/y) ^(A)
Dewing	CS	12	0	$80 \rightarrow 25$	saturated	0.15
	1Cr	12	0	$80 \rightarrow 25$	saturated	0.12
	3Cr	12	0	$80 \rightarrow 25$	saturated	0.07
	CS	12	200	$80 \rightarrow 25$	saturated	0.82
	1Cr	12	200	$80 \rightarrow 25$	saturated	0.76
	3Cr	12	200	$80 \rightarrow 25$	saturated	0.42

 TABLE 5

 Summary of Corrosion Rate Data of Three Different Steels in CO₂ Phase

^(A) Standard deviation of corrosion rate was within 5%.







FIGURE 11. SEM and EDS surface analyses of the corrosion product on the sample surface after corrosion experiment in CO_2 phase experiencing temperature fluctuation without H_2S : (a) CS, (b) 1Cr steel, and (c) 3Cr steel.



FIGURE 12. Low magnification SEM images of the sample surface after corrosion experiment in CO_2 phase experiencing temperature fluctuation with 200 ppm of H_2S : (a) CS, (b) 1Cr steel, and (c) 3Cr steel.



FIGURE 13. SEM and EDS surface analyses of the sample surface after corrosion experiment in CO_2 phase experiencing temperature fluctuation with 200 ppm of H_2S : (a) CS, (b) 1Cr steel, and (c) 3Cr steel.





FIGURE 14. SEM images and EDS spectra of the cross section of samples exposed to CO_2 phase experiencing temperature fluctuation with 200 ppm of H_2S : (a) CS and (b) 3Cr steel.

under this condition. Furthermore, as shown in Figure 14, in the case of 3Cr steel, a thinner layer was observed with a Cr-enriched inner layer compared with CS. This Cr-enriched inner layer could contribute to the reduction of the corrosion rate of 3Cr steel.

It is clearly shown from these results that the presence of H_2S alters the water condensation behavior on the steel surface. It makes a drop of water spread out and form a thin water layer, which is consistent with the results in CO_2 phase without condensation. Furthermore, the observation of uniform and continuous inner FeCO₃ layer confirms the presence of a thin water layer on the steel surface that becomes rapidly supersaturated with FeCO₃, leading to FeCO₃ formation.

CONCLUSIONS

The corrosion properties of pipeline steels in $CO_2/H_2S/H_2O$ mixtures with different amounts of water (both saturated and undersaturated) were

investigated by weight-loss measurements and surface analysis techniques. The following conclusions are drawn:

* There was no significant corrosion attack in the supercritical and liquid CO_2 phases in the presence of water (both saturated and undersaturated).

♦ The addition of 200 ppm H_2S in the CO₂ phase dramatically increased the corrosion rate of all tested materials (CS, 1Cr, and 3Cr steels) when CO₂ was saturated with water.

Under dewing conditions, corrosion occurs both in pure CO₂ and CO₂/H₂S systems as a result of the formation of water droplets/layer on the sample surface.
 3Cr steel showed better corrosion resistance for the tested conditions compared with CS and 1Cr steel.

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