



## Investigating the Effect of H<sub>2</sub>S and Corrosion Inhibitor on the Corrosion of Mild Steel under High Pressure CO<sub>2</sub> Conditions

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### ABSTRACT

The effect of H<sub>2</sub>S and corrosion inhibitor on the aqueous corrosion behavior of mild steel was evaluated at high CO<sub>2</sub> partial pressure conditions. The experiments were performed in a 7.5 L autoclave with different temperatures (25°C and 80°C) and different H<sub>2</sub>S concentrations (1000 ppm<sub>v</sub> and 2000 ppm<sub>v</sub>) at 12 MPa CO<sub>2</sub>. The corrosion rate of steel samples was determined by electrochemical and weight loss measurements. The surface and cross-sectional morphology and the composition of the corrosion product layers were analyzed by using surface analytical techniques (SEM, EDS and XRD). Results showed that the presence of 1000 ppm<sub>v</sub> and 2000 ppm<sub>v</sub> H<sub>2</sub>S decreased the corrosion rate of mild steel compared with pure CO<sub>2</sub> condition. However, the final corrosion rates were still higher than the targeted threshold (< 0.1 mm/y). Surface and cross-sectional analyses revealed the formation of FeS in the presence of H<sub>2</sub>S and no localized corrosion rate below 0.1 mm/y in high pressure CO<sub>2</sub> conditions with 2000 ppm<sub>v</sub> H<sub>2</sub>S.

Key words: Supercritical CO<sub>2</sub>, CO<sub>2</sub> corrosion, carbon steel, H<sub>2</sub>S, corrosion inhibitor

### INTRODUCTION

Over the past decade, there has been increasing interest in the corrosion behavior of carbon steels in supercritical  $CO_2$  conditions. Unlike the case of carbon capture and storage (CCS) where small amounts of water are present, the exploitation of fields with high pressures of  $CO_2$  needs to consider the presence of formation water, which presents strong corrosivity. It has been reported that the aqueous corrosion rate of carbon steel at high  $CO_2$  pressures (liquid and supercritical  $CO_2$ ) without protective FeCO<sub>3</sub> corrosion product layers is very high (>20 mm/y) due to the high concentrations of corrosive species such

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as H<sup>+</sup> and H<sub>2</sub>CO<sub>3</sub>.<sup>1-5</sup> Steels with low Cr contents (i.e., 1% Cr and 3% Cr) have shown no beneficial effect in terms of reducing the corrosion rate to admissible values.<sup>6</sup> Therefore, controlling corrosion in these cases usually involves the use of corrosion resistant alloys (CRAs) or corrosion inhibitors (CI). Adequate protection of carbon steel was achieved by applying CI in high pressure CO<sub>2</sub> environments.<sup>6</sup>

The presence of small amounts of H<sub>2</sub>S in high pressure CO<sub>2</sub> environments greatly alters the corrosion behavior of carbon steel.<sup>6-10</sup> For example, in the presence of 200 ppm<sub>v</sub> H<sub>2</sub>S, the corrosion rate of carbon steel was reduced by about 10 times compared to the case of pure CO<sub>2</sub> at 12 MPa CO<sub>2</sub> and 80°C, but corrosion rate was still high and additional protection was required. In addition, the corrosion resistance of low Cr steels (1% Cr and 3% Cr) was worse than that of carbon steel in the above conditions. On the other hand, the addition of an imidazoline based CI in a high pressure  $CO_2$  environment with 200 ppm<sub>y</sub> H<sub>2</sub>S reduced the corrosion rate of carbon steel below 0.1 mm/y.<sup>6</sup> The effect of higher H<sub>2</sub>S concentrations on corrosion rate of carbon steel with and without inhibitor addition has not been thoroughly studied vet.

The objective of the present study was to increase the understanding of the effect of H<sub>2</sub>S and CI on the aqueous corrosion behavior of mild steel under high pressure CO<sub>2</sub> environments with elevated H<sub>2</sub>S concentrations up to 2000 ppmy.

#### **EXPERIMENTAL PROCEDURE**

The test specimens were machined from UNS K03014<sup>(1)</sup> carbon steel (CS) with two different geometries: cylindrical type with 5 cm<sup>2</sup> exposed area for electrochemical measurements, and rectangular type with a size of 1.27 cm × 1.27 cm × 0.254 cm for surface analysis. The detailed chemical composition of the steel is shown elsewhere.<sup>6,11</sup> The specimens were ground sequentially with 250, 400 and 600-grit silicon carbide paper, rinsed with deionized (DI) water, cleaned with isopropyl alcohol in an ultrasonic bath for 60 seconds, and then dried.

An imidazoline generic CI was selected for evaluation under high pCO<sub>2</sub> environments with H<sub>2</sub>S. In this instance, 'imidazoline' is shorthand for tall oil fatty acid (TOFA) imidazoline-based inhibitor. The detailed chemical composition of the CI is found in our previous work.<sup>6,12</sup>

The corrosion experiments were conducted in a 7.5-liter autoclave (UNS N10276) which contained a CS working electrode, a HPHT Ag/AgCI reference electrode, and a platinum-coated niobium counter electrode. The test solution was a 1 wt.% NaCl aqueous electrolyte prepared using DI water. An impeller was used to stir the solution and to generate flow velocities of about 1 m/s (at 1000 rpm) during the tests.

Corrosion rate and corrosion potential of specimens were evaluated with time by linear polarization resistance (LPR) measurements. After each test, the morphology and compositions of corrosion products were analyzed with scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). Average corrosion rates were also determined from the weight-loss (WL) method. In addition, attack morphology was examined after corrosion product removal and cleaning of the samples with Clarke's solution.

The details of the experimental procedure for the autoclave setup and corrosion rate measurements are shown in our previous work.<sup>6,11</sup> Table 1 shows the test conditions used in the present study. All experiments were conducted within 3 days, in order to minimize the effect of the solution contamination due to corrosion reaction over time.

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Conditions for corrosion tests			
pCO <sub>2</sub> (MPa)	H <sub>2</sub> S (ppm <sub>v</sub> )	Temperature (°C)	CI (ppm <sub>v</sub> )
12	1000	25 and 80	0
12	2000	25 and 80	0
12	2000	25 and 80	400
12	2000	80	200

Table 1 Conditions for corrosion tests

#### **RESULTS AND DISCUSSION**

#### Experiments at 12 MPa CO2 and 25°C

Figure 1 shows LPR and WL corrosion rate data of CS exposed at 12 MPa CO<sub>2</sub> and 25°C with 1000  $ppm_v$  and 2000  $ppm_v$  of H<sub>2</sub>S. Corrosion rates seemed to reach stable values after 20 hours of exposure for both H<sub>2</sub>S concentrations. It is worth noting that increasing the H<sub>2</sub>S concentration led to higher corrosion rates in this experimental condition. WL corrosion rates showed the same trend as the LPR corrosion rates.



# Figure 1: Corrosion rate of CS with different $H_2S$ concentrations at 12 MPa CO<sub>2</sub> and 25°C: (a) LPR corrosion rate, (b) WL corrosion rate.

Figure 2 shows the surface morphology and chemical analysis of the corrosion product formed at 12 MPa  $CO_2$  and 25°C with 1000 ppm<sub>v</sub> H<sub>2</sub>S. The corrosion product was mostly uniform and presented some discontinuous cracks due to dehydration. The chemical analysis performed by EDS at the top of the corrosion product indicated that it consisted mainly of iron and sulfur.

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Figure 2: SEM surface view (a) and EDS analysis (b) of the corrosion product formed at 12 MPa  $CO_2$  and 25°C with 1000 ppm<sub>v</sub> H<sub>2</sub>S.

Figure 3 displays the SEM cross-sectional view and EDS elemental mapping of the corrosion product layer formed at 12 MPa CO<sub>2</sub> and 25°C with 1000 ppm<sub>v</sub> H<sub>2</sub>S. It revealed a formation of thin and mainly continuous corrosion product layer (~10 µm) which was not very protective because of the relatively high corrosion rate of ~2 mm/y seen at the end of the experiment. This layer mainly consisted of iron and sulfur indicating the formation of FeS.



# Figure 3: SEM cross-sectional view and EDS elemental mapping analysis of the corrosion product formed at 12 MPa CO<sub>2</sub> and 25°C with 1000 ppm<sub>v</sub> H<sub>2</sub>S.

The corrosion product formed with 2000 ppm<sub>v</sub> H<sub>2</sub>S (Figure 4) looked more porous than that of the sample exposed to 1000 ppm<sub>v</sub> H<sub>2</sub>S, and also seemed less adherent and and fragile when corroded samples where manipulated. This would explain the higher corrosion rates measured for the higher concentration of H<sub>2</sub>S. Corrosion products containing iron and sulfur as main elements were also observed in the case with 2000 ppm<sub>v</sub> H<sub>2</sub>S. The cross-sectional analysis (Figure 5) showed a porous layer of ~10 µm thickness composed mainly of iron and sulfur.

Figure 6 shows the surface morphologies of corroded samples after corrosion product removal. For both conditions, surfaces were relatively rough but no significant pits or localized attack was observed and the attack was considered as uniform.

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Figure 4: SEM surface view (a) and EDS analysis (b) of the corrosion product formed at 12 MPa  $CO_2$  and 25°C with 2000 ppm<sub>v</sub> H<sub>2</sub>S.



Figure 5: SEM cross-sectional view and EDS elemental mapping of the corrosion product formed at 12 MPa CO<sub>2</sub> and 25°C with 2000 ppm<sub>v</sub> H<sub>2</sub>S.



Figure 6: SEM images of corroded surfaces after corrosion product removal: (a) 12 MPa CO<sub>2</sub>, 25°C, 1000 ppm<sub>v</sub> H<sub>2</sub>S, (b) 12 MPa CO<sub>2</sub>, 25°C, 2000 ppm<sub>v</sub> H<sub>2</sub>S.

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#### Experiments at 12 MPa CO2 and 80°C

Figure 7 shows LPR and WL corrosion rate data of carbon steel at 12 MPa CO<sub>2</sub> and 80°C with different H<sub>2</sub>S concentrations. Corrosion rates seemed to attain stable values after about 20 hours and 35 hours of exposure for 1000 ppm<sub>v</sub> and 2000 ppm<sub>v</sub> H<sub>2</sub>S concentrations, respectively. Similar to the results at 25°C, increasing the H<sub>2</sub>S concentration led to higher corrosion rates at 80°C. The initial corrosion rate with 1000 ppm<sub>v</sub> H<sub>2</sub>S was 10.6 mm/y and slowly decreased with time to 6.4 mm/y after 48 hours. For 2000 ppm<sub>v</sub> H<sub>2</sub>S, the initial corrosion rate was ~11 mm/y and increased to ~16 mm/y after 9 hours, then it decreased to 8.5 mm/y and remained somewhat stable until the end of the experiment. The corrosion rates obtained by WL measurements showed good agreement with the LPR measurements (average).

![](_page_5_Figure_2.jpeg)

Figure 7. Corrosion rate of CS with different  $H_2S$  concentrations at 12 MPa CO<sub>2</sub> and 80°C: (a) LPR corrosion rate, (b) WL corrosion rate.

Figure 8 shows the surface morphology and chemical analysis for a sample exposed at 12 MPa CO<sub>2</sub> and 80°C with 1000 ppm<sub>v</sub> H<sub>2</sub>S. The corrosion product was mostly uniform and free of cracks, and some scattered corrosion products were observed on top of the main corrosion product layer. The EDS analysis showed the presence of iron and sulfur as main constituents.

![](_page_5_Figure_5.jpeg)

Figure 8: SEM surface view (a) and EDS analysis (b) of the corrosion product formed at 12 MPa  $CO_2$  and 80°C with 1000 ppm<sub>v</sub> H<sub>2</sub>S.

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Figure 9 shows a SEM cross sectional view and EDS elemental mapping of the corrosion product shown in Figure 8. A relatively thick and continuous layer of approximately 40 µm thickness was observed. This layer did not seemed to be well adhered to the steel surface which could explain the high corrosion rate measured at this experimental condition. The elemental mapping showed mainly iron and sulfur suggesting the presence of FeS. Some oxygen close to the steel surface was also observed probably due to the formation of an iron oxide. It is interesting to note that a very thin (few microns) nickel rich layer was detected at the very top of the corrosion product layer. This layer is not believed to have affected the corrosion process and it could have formed from foreign nickel ions provenient from the used Hastelloy autoclave.

![](_page_6_Picture_1.jpeg)

# Figure 9: SEM Cross-sectional view and EDS elemental mapping of the corrosion product formed at 12 MPa CO<sub>2</sub> 80°C with 1000 ppm<sub>v</sub> H<sub>2</sub>S.

Figure 10 shows the morphology and chemical analysis of the corrosion product on carbon steel samples exposed at 12 MPa CO<sub>2</sub> and 80°C with 2000 ppm<sub>v</sub> H<sub>2</sub>S. The corrosion product was mainly uniform with some cracks due to dehydration; and some well defined scattered crystals were observed on top of the corrosion product layer. The EDS analysis showed that both the corrosion product layer and the crystals mainly consisted of iron and sulfur, indicating the formation of FeS.

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The cross-sectional analysis, presented in Figure 11, shows that the corrosion product layer had a thickness ~130 µm including the crystals on top. Both top and bottom parts of the layer showed a porous structure and the corrosion product was poorly adhered to the steel surface, which is in line with the high corrosion rate seen on these samples. The elemental mapping (Figure 11) clearly shows that the layer consisted of iron and sulfur. A complementary XRD analysis (Figure 12) corroborated the presence of FeS (Mackinawite). It is interesting to note that iron carbonate (FeCO<sub>3</sub>) was not detected on the surface of samples exposed to both 1000 ppm<sub>v</sub> and 2000 ppm<sub>v</sub> of H<sub>2</sub>S. This would indicate that under the tested conditions, the kinetics of formation of FeS is much faster than that of FeCO<sub>3</sub> regardless of the high used partial pressure of CO<sub>2</sub> (12 MPa).

Figure 13 shows the surface morphologies of samples after corrosion product removal. For both conditions, corroded surfaces were considerably rough but no significant localized damage was found and the attack was considered as uniform.

![](_page_7_Figure_2.jpeg)

Figure 10: SEM surface view (a) and EDS analysis (b) of the corrosion product formed at 12 MPa  $CO_2$  and 80°C with 2000 ppm<sub>v</sub> H<sub>2</sub>S.

![](_page_7_Picture_4.jpeg)

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![](_page_8_Figure_0.jpeg)

Figure 11: SEM Cross-sectional view and EDS elemental mapping of the corrosion product formed at 12 MPa CO<sub>2</sub> and 80°C with 2000 ppm<sub>v</sub> H<sub>2</sub>S.

![](_page_8_Figure_2.jpeg)

Figure 12: XRD surface analysis of a CS sample exposed at 12 MPa CO<sub>2</sub> and 80°C with 2000 ppm<sub>v</sub>.

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![](_page_9_Picture_0.jpeg)

Figure 13: SEM images of corroded surfaces of samples after corrosion product removal: (a) 12 MPa CO<sub>2</sub>, 80°C, 1000 ppm<sub>v</sub> H<sub>2</sub>S, (b) 12 MPa CO<sub>2</sub>, 80°C, 2000 ppm<sub>v</sub> H<sub>2</sub>S.

#### Experiments at 12 MPa CO<sub>2</sub> and 2000 ppm<sub>v</sub> H<sub>2</sub>S with CI

As a summary, Figure 14 shows the change of corrosion rate of carbon steel with different  $H_2S$  concentrations (from 0 to 2000 ppm<sub>v</sub>) at 25°C and 80°C under 12 MPa CO<sub>2</sub> condition. The corrosion rates with  $H_2S$  concentrations <1000 ppm<sub>v</sub> were taken from a previous study.<sup>6</sup> It can be clearly seen that the addition of small amounts of  $H_2S$  reduced the corrosion rate in the presence of high partial pressure of CO<sub>2</sub>. However, these reduced corrosion rates were still high and additional protection was required to achieve admissible values (i.e., < 0.1 mm/y). Thus, a series of tests with addition of CI were carried out with the aim of reducing the corrosion rate of carbon steel in the high pressure CO<sub>2</sub> environment with 2000 ppm<sub>v</sub>  $H_2S$ . A CI concentration of 400 ppm<sub>v</sub> was selected based on previous results in high pressure CO<sub>2</sub> environments with and without 200 ppm<sub>v</sub>  $H_2S$ .

![](_page_9_Figure_4.jpeg)

## Figure 14: Change of corrosion rate of CS with H<sub>2</sub>S concentrations at different temperatures under 12 MPa CO<sub>2</sub> condition (48-62 hours of exposure).

The corrosion behavior of carbon steel with 400  $ppm_v$  of CI in environments with 12 MPa CO<sub>2</sub> and 2000  $ppm_v$  H<sub>2</sub>S at 25°C and 80°C is shown in Figure 15. It is seen that 400  $ppm_v$  of CI reduced the corrosion

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rate to values lower than 0.1 mm/y for both temperatures. This effect was more pronounced at  $25^{\circ}$ C, where corrosion rates stabilized at ~0.002 mm/y, while corrosion rates at 80°C seemed to stabilize at ~0.09 mm/y.

![](_page_10_Figure_1.jpeg)

# Figure 15: Corrosion rate for CS with 400 ppm<sub>v</sub> of CI at 12 MPa CO<sub>2</sub> and 2000 ppm<sub>v</sub> H<sub>2</sub>S (25°C and 80°C).

Surface analysis of the samples exposed with 400  $ppm_v$  CI (Figure 16) showed no visible corrosion attack for both tested temperatures.

![](_page_10_Figure_4.jpeg)

# Figure 16: SEM images of the surfaces of samples exposed with 400 ppm<sub>v</sub> of CI at 12 MPa CO<sub>2</sub> and 2000 ppm<sub>v</sub> H<sub>2</sub>S: (a) 25°C, (b) 80°C.

Since the final corrosion rate of carbon steel exposed at 80°C with 400 ppm<sub>v</sub> of CI was close to the admissible value of 0.1 mm/y (Figure 15b), it was decided to perform an additional test with a lower CI concentration of 200 ppm<sub>v</sub> to check if corrosion rate could increase significantly above the targeted threshold.

Figure 17 shows the corrosion behavior of carbon steel with 200 ppm<sub>v</sub> of CI at 12 MPa  $CO_2$  and 80°C with 2000 ppm<sub>v</sub> H<sub>2</sub>S. The initial corrosion rate was low at about 0.02 mm/y. However, it slowly increased

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with time to 0.13 mm/y after 66 hours, indicating that 200 ppm<sub>v</sub> of CI was not sufficient to reduce corrosion rate below the targeted value of 0.1 mm/y under the present conditions. A significant amount of corrosion products were found on the surface of the sample exposed with 200 ppm<sub>v</sub> of CI at 80°C as shown in Figure 18. These corrosion products contained mainly iron and sulfur suggesting the formation of FeS.

![](_page_11_Figure_1.jpeg)

Figure 17: Variation of corrosion rate for CS with 200 ppm $_{v}$  CI at 12 MPa CO<sub>2</sub> and 80°C with 2000 ppm $_{v}$  H<sub>2</sub>S.

![](_page_11_Figure_3.jpeg)

Figure 18: SEM surface view and EDS analysis of the corrosion product formed with 200 ppm<sub>v</sub> CI at 12 MPa CO<sub>2</sub> and 80°C with 2000 ppm<sub>v</sub> H<sub>2</sub>S.

### CONCLUSIONS

The effect of  $H_2S$  (up to 2000 ppm<sub>v</sub>) and CI on the aqueous corrosion behavior of carbon steel was evaluated at high pressure (12 MPa) CO<sub>2</sub> conditions at 25°C and 80°C. The following conclusions are drawn:

 The presence of 1000 ppm<sub>v</sub> and 2000 ppm<sub>v</sub> of H<sub>2</sub>S decreased the corrosion rate of carbon steel. However, the final corrosion rates were still higher than the targeted admisible value of 0.1 mm/y; thus, the addition of corrosion inhibitor was needed to control corrosion.

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- The corrosion rate increased with increasing H<sub>2</sub>S concentration from 1000 ppm<sub>v</sub> to 2000 ppm<sub>v</sub>. The corrosion product layers were found to be formed mainly of FeS (Mackinawite).
- No localized corrosion was observed with 1000 ppm<sub>v</sub> and 2000 ppm<sub>v</sub> H<sub>2</sub>S under the tested high pCO<sub>2</sub> condition.
- The addition of 400 ppm<sub>v</sub> of imidazoline-based corrosion inhibitor suscessfully reduced the corrosion rate of carbon steel below 0.1 mm/y at 25°C and 80°C in environment with 12 MPa CO<sub>2</sub> and 2000 ppm<sub>v</sub> H<sub>2</sub>S. Lower inhibitor concentrations are not recommended at 80°C.

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