



#### Effect of Elevated Temperatures (30-80 °C) on CO<sub>2</sub> Corrosion after Intermittent Oil/Water Wetting

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#### ABSTRACT

The main goal of this research is to electrochemically determine the influence of temperature elevation (30 °C - 80 °C) on the wetting state and corrosion behavior of mild steel during intermittent oil/water wetting. The results obtained in separate experiments demonstrated that the corrosion mitigation effect of an oil phase (LVT-200 model oil) containing myristic acid (surface-active compound representing naturally occurring oxygen-containing compounds in crude oils) on  $CO_2$  corrosion decreased with temperature after intermittent wetting. The influence of the same temperature range on interfacial properties during intermittent oil/water wetting such as oil/water interfacial tension (IFT) and oil-in-water contact angle were also investigated. For model oil containing myristic acid, the IFT value was unchanged with temperature and could not be correlated to the corrosion rate. In the presence of the oil phase, the surface became more hydrophilic at 55 °C, which diminished the adsorption and persistency of the oil layer and decreased the corrosion mitigation effect. The effect of temperature on IFT and contact angle can depend on the nature of oil and surface-active compound.

Key words: CO<sub>2</sub> corrosion, carbon steel, oil/water intermittent wetting, naturally occurring surface-active compound, elevated temperatures.

#### INTRODUCTION

Carbon steel pipelines are the most efficient and economical method for transporting oil and natural gas, especially for their large-scale transfer across long distances<sup>1,2</sup>. However, these pipelines are prone to corrosion due to the presence of corrosive gases such as carbon dioxide (CO<sub>2</sub>) and/or hydrogen sulfide (H<sub>2</sub>S) dissolved in any present aqueous phase derived from reservoir brine. The oil phase itself does not lead to corrosion and can even be preventive<sup>3,4,5,6</sup>. In oil-water flow through a horizontal pipeline at low flow rates, the aqueous phase is present as a separate layer flowing at the bottom of the pipe. This corresponds to a stratified flow regime because gravity dominates over turbulent forces. By increasing the flow rate inside the pipeline, the turbulent energy increases, as a result the water phase can become gradually dispersed as droplets in the oil phase <sup>7, 8,9</sup>. When considering pipelines operated at low water cut, the water phase is typically entrained by the hydrocarbon phase and the oil phase wets the internal surface of the pipe, minimizing the risk of corrosion. By increasing the water content, water breakout may occur, consequently water may wet more easily the surface of the pipe, and corrosion can become possible<sup>10,11,12</sup>. Considering that production flow rates can vary significantly during the life of a pipeline

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system, different flow patterns can be expected in which oil and water can alternately wet the pipe internal surface. This phenomenon is identified as "intermittent wetting"<sup>13</sup>, flow rate and water cut playing governing factors thereon<sup>12</sup>. This influences the mechanism of the corrosion of the metal surface which may differ from those commonly accepted for aqueous corrosion (i.e., full water wetting) <sup>3,7,14,15</sup>. The wetting condition of the metal surface is consequently a key parameter to predict corrosion behavior<sup>16,17,18,19</sup>, and it is important to know the relation between surface wetting and corrosion processes<sup>20,21,22</sup>. This knowledge can help decrease economic costs and mitigate the potential for adverse environmental impacts caused by corroding tubular steels. Although the corrosion behavior of carbon steel during intermittent wetting conditions at low temperature has been investigated in our previous studies <sup>23</sup>, little has been reported on the mitigation effect of oil/water intermittent wetting at elevated temperatures.

Crude oils can be defined as naturally occurring liquid mixtures of hydrocarbons (83 wt.%–87 wt.% of carbon, 10 wt.%–14 wt.% of hydrogen) containing derivatives of nitrogen (0.1 wt.%–2.0 wt.%), oxygen (0.05 wt.%–1.5 wt.%), sulfur (0.05 wt.%–6.0 wt.%), metals (less than 1000 ppm), and other elements<sup>24,25</sup>. Some chemical compounds native to crude oils identified as surface-active compounds have an ability to preferentially adsorb at steel-water, steel-oil, and oil-water interfaces, which can alter the wetting and corrosion properties of steel surfaces<sup>7</sup>. Surface-active compounds such as oxygen compounds, sulfur compounds, and nitrogen compounds were identified as factors influencing the wettability preference of a crude oil <sup>26</sup>. The corrosion inhibition and wettability effect of oxygen containing compounds has been studied <sup>20,22</sup>. In this study, the effect of a select carboxylic acid surface-active compound (myristic acid) on corrosion inhibition, interfacial tension, steel/oil contact angle, and oil/water interface structure is described for elevated temperatures.

#### EXPERIMENTAL PROCEDURE

#### **Corrosion rate measurements**

#### **Chemicals and Materials**

In this study, LVT-200<sup>‡</sup> model oil, which is a hydrotreated light distillate petroleum fraction<sup>27</sup>, was used with and without the addition of 0.1 wt.% of myristic acid, which is representative of oxygen containing compounds that naturally exist in crude oils. The surfactant concentrations used in this study are in the common range for such a type of naturally occurring compound in crude oils. The molecular structure of myristic acid is shown in Figure 1. Note the amphiphilic character of myristic acid.



Figure 1. Molecular structure of myristic acid

The metal specimens used for electrochemical measurements were machined from a X65 carbon steel, a typical pipeline steel material used upstream in the oil and gas industry. This X65 carbon steel has a uniform, fine structure of cementite in a ferrite matrix and conforms to applicable specifications for API

<sup>&</sup>lt;sup>‡</sup> Trade Name

С	Mn	Nb	Р	S	Ti	V	Fe
0.05%	1.40%	0.04%	0.003%	<0.001%	0.01%	0.03%	balance

#### Table 1. Chemical compositional analysis of X65 pipeline steel

#### Experimental Apparatus and Procedure

Experiments were performed in a standard three electrode glass cell with an X65 carbon steel rotating cylinder electrode (RCE) working electrode, a platinum-coated titanium mesh counter electrode, and an Ag/AgCl (KCl saturated) reference electrode. Before each experiment, the RCE was sequentially polished with 400 and 600 grit silicon carbide abrasive papers, cleaned with isopropanol in an ultrasonic bath, and air-dried. Both oil and water solutions were deoxygenated and saturated with carbonic species by 1 hour of sparging with CO<sub>2</sub> before the introduction of the working electrode. After the RCE was inserted into the glass cell, a pre-corrosion test was conducted to determine whether the initial corrosion rate was close to the blank experiment corrosion rate to ensure no contamination occurred from the previous test. Linear polarization resistance (LPR) measurements were taken with a scan range from -5 mV to +5 mV vs. OCP, a scan rate of 0.125 mV/s, and a B value of 26 mV using a Gamry<sup>§</sup> Interface 1010B<sup>™</sup> potentiostat. The glass cell was sparged with CO<sub>2</sub> throughout the test to prevent air (oxygen) ingress and to maintain saturation of the aqueous electrolyte with CO<sub>2</sub>. To minimize the noise in electrochemical measurements caused by CO<sub>2</sub> sparging, the sparge tube was retracted into the headspace during data acquisition. The pH was adjusted by adding deoxygenated hydrochloric acid or sodium bicarbonate solution during each experiment as needed. The experiments were performed using intermittent wetting procedure, where the electrochemical glass cell was connected to another reservoir glass cell to change the level of the water phase in the main glass cell, enabling placement of the specimen in oil and water, alternately. These two glass cells are connected to each other using a pump with two solenoid valves. A simple schematic of the intermittent wetting setup is shown in Figure 2. Experiments were performed therein in four steps:

- 1. Pre-corrosion: polished specimen was immersed in the water phase.
- 2. Partitioning: 300 ml of an oil layer containing surface-active compounds was added to the top of the water phase. The polished specimen was still immersed into the water phase and rotated for one hour at 1000 rpm.
- 3. Intermittent wetting: The intermittent wetting step includes two sub-steps:
  - a. Oil wetting step: Brine is pumped from the electrochemical cell to the reservoir cell to lower the oil layer, so the oil layer completely envelopes the specimen surface.
  - b. Water wetting step: Brine is pumped from the reservoir cell back to the electrochemical cell to lift the oil layer well above the specimen surface.
- 4. Persistency: After the intermittent wetting cycles, the specimen was returned to the water phase. The corrosion rate was measured every 20 minutes for at least 10 hours.

A schematic picture of the procedure for these experiments is shown in Figure 3. The test matrix used to study the effect of elevated temperatures on corrosion rate after intermittent contact with oil is presented in Table 2.

<sup>§</sup> Trade Name



Figure 2. the intermittent wetting setup



Figure 3. Procedure of intermittent wetting experiments: a) pre-corrosion; b) partitioning; c) oil/water intermittent wetting repeated multiples times over 1 hour; and d) persistency.

Parameter	Conditions
Material	X65 steel
Electrolyte	1 wt.% NaCl in DI water
Oil phase	100% model oil (15% of total liquid)
Temperature	30,55,80 °C
CO <sub>2</sub> partial pressure	0.96, 0.84, 0.52 bar
рН	4.0
RCE rotational speed	1000 rpm
Intermittent wetting period	1 minute
Duration of intermittent wetting	1 hour
Surface-active compound	Myristic acid (0.1, 0.5, 1, 2 wt.% of oil phase)
Experimental equipment	RCE, Glass cell
Electrochemical measurements	OCP, LPR, EIS

#### Interfacial tension measurements

Interfacial tension is the measure of free energy per unit area of interface between two liquid phases, which can help to characterize changes that occur at the oil/water interfaces <sup>28,29</sup>. Oil-water interfacial tension measurements were performed to evaluate the surface activity of acidic compounds. The measurements were done using the K20 KRÜSS<sup>\*\*</sup> tensiometer and Wilhelmy plate method. Both 1 wt.%

<sup>\*\*</sup> Trade Name

NaCl solution and model oil containing surface-active compounds were sparged with  $CO_2$  for at least 1 hour. Temperature and pH of both were adjusted to the same values as for electrochemical characterization before each measurement. Measurements were performed with 30-50 ml of each phase. Each measurement was repeated at least three times.

#### Contact angle measurements

Contact angle ( $\theta$ ) can be used to measure wettability, which is most often described by the geometry of a sessile or resting drop (Figure 4). When water is one of the liquids, a low contact angle ( $\theta < 90^{\circ}$ ) indicates hydrophilicity, and a high contact angle ( $\theta > 90^{\circ}$ ) indicates hydrophobicity<sup>30</sup>. The oil-in-water contact angle measurements were done using a goniometer system designed built by Tang<sup>30</sup>, which is shown in Figure 5. The oil-in-water contact angle simulates the water pre-wet condition which is representative of pre-wetting condition of the corrosion rate experiments.



Figure 4. Sessile drop on a solid surface, surrounded by either another liquid, gas or vapor<sup>30</sup>.



Figure 5. Photograph of the goniometer system with goniometer cell, backlight and a camera<sup>30</sup>.

#### RESULTS

#### Effect of elevated temperatures on corrosion rates

The results for baseline corrosion rate experiments at pH 4.0, 30 and 55  $^{\circ}$ C, 0.96 bar CO<sub>2</sub>, 1000 rpm are shown in

Figure **6**. The baseline experiment shows the corrosion rate of mild steel in the presence of only the water phase, i.e., no oil phase was present in the glass cell. A stable corrosion rate was obtained of 4 mm/year at 30 °C, increasing to 7 mm/year at 55 °C. The potentiodynamic sweeps for baseline experiments at 30

and 55 °C, shown in Figure 7, indicate that the cathodic and anodic reactions accelerated with temperature. The results for corrosion rate experiments, which also experienced an intermittent oil/water wetting condition using model oil containing different concentrations of myristic acid at 55 °C, are shown in Figure 8. In the 'intermittent wetting' step of these experiments, the specimen was periodically immersed in each phase for one minute and this cycle was repeated for one hour. It was observed that the corrosion rate did not change in the partitioning step, however, a significant decrease was measured after the intermittent wetting cycles. The persistency of this decrease in the corrosion rate was dependent on the myristic acid concentration. For 0.1 wt.% myristic acid concentration, the mitigation effect due to intermittent wetting was not persistent and the corrosion rate was gradually increasing until it reached the baseline corrosion rate. For 1 wt.% myristic acid concentration, the corrosion rate decreased to 1.8 mm/year which indicates that the mitigation effect was increased by myristic acid concentration. However, the corrosion rate increased to 2.5 mm/year after approximately 5 hours. This shows that the structure of the oil and surfactant layer formed on the steel specimen has changed over time. After that increase, a stable corrosion rate was observed for at least 10 hours. The same behavior was observed for 2 wt.% myristic acid concentration, except a lower final corrosion rate value was obtained which shows a stronger mitigation effect compared to lower myristic acid concentrations. Comparing intermittent wetting results at 55 °C to those at 30 °C, which are shown in Figure 9, it can be seen that the corrosion mitigation effect of the oil layer formed on the specimen surface decreases with temperature.



Figure 6. Baseline corrosion rates at 30 and 55 °C, 0.96 bar CO<sub>2</sub>, pH 4.0, and 1000 rpm.



Figure 7. Potentiodynamic sweeps at 30 and 55 °C, pH 4.0, and 1000 rpm.



Figure 8. Corrosion rate in the presence of model oil containing 0.1, 1, 2 wt.% myristic acid at pH 4.0, 55 °C, 0.84 bar CO<sub>2</sub> and 1000 rpm for one hour intermittent wetting.



Figure 9. Corrosion rate in the presence of model oil containing 0.1, 0.5, 1 wt.% myristic acid at pH 4.0, 30 °C, 0.96 bar CO<sub>2</sub> and 1000 rpm for one hour intermittent wetting.

The results for baseline corrosion rate experiments at pH 4.0, 30 to 80 °C, 0.96 bar CO<sub>2</sub>, 1000 rpm are shown in Figure 10. The stable corrosion rate increased to 8.5 mm/year at 80 °C. The potentiodynamic sweeps for baseline experiments at 30, 55 and 80 °C, which are shown in Figure 11, indicate that both cathodic and anodic reactions were accelerated with temperature. The corrosion rate results for intermittent oil/water wetting experiments using model oil containing different concentrations of myristic acid at 80 °C are shown in Figure 12. The oil/water intermittent wetting with 0.1 and 1 wt.% myristic acid concentration in the oil phase showed no mitigation effect on the corrosion rate.



Figure 10. Baseline corrosion rates for 30, 55 and 80°C at pH 4.0, 1000 rpm.



Figure 11. Potentiodynamic sweeps at 30 and 55 °C for pH 4.0, 1000 rpm.



Figure 12. Corrosion rate in the presence of model oil containing 0.1, 1 wt.% myristic acid at pH 4.0, 80 °C, 0.52 bar CO<sub>2</sub> and 1000 rpm for one hour intermittent wetting.

Figure 13 shows a comparison of corrosion rates for baseline and oil/water intermittent wetting experiments. The mitigation effect decreased at 55 °C as compared to 30 °C. Moreover, the mitigated corrosion rates obtained right after the intermittent wetting step were not stable, and increased until they reached a stable value. At 80 °C, there was no effect of the presence of the oil phase and intermittent

wetting even with a high concentration of 1 wt.% myristic acid. Overall, it was observed that the corrosion mitigation effect of the oil layer decreased with temperature.



# Figure 13. Corrosion rate for baseline experiments and intermittent wetting experiments in the presence of model oil containing 0.1, 1 wt.% myristic acid at 30, 55 and 80°C (corresponding to 0.96, 0.84 and 0.52 bar CO<sub>2</sub>), pH 4.0, and 1000 rpm.

#### Effect of elevated temperatures on oil-water interfacial tension

According to the corrosion rate results, which showed that the corrosion mitigation effect of the oil film decreases with temperature, a possible mechanism is proposed based on the structure of the film formed at the oil/water interface to explain the effect of elevated temperatures on corrosion behavior of the X65 steel. Schematics of the postulated structures related to the film formed at these two interfaces are shown in

**Figure 14**. It can be postulated that fewer surface-active molecules adsorb to the oil-water interface at higher temperature, leading to an increase in the interfacial tension value, a less ordered and less protective surfactant layer. In order to investigate the proposed mechanism, the interfacial tensions between oil/water interfaces were measured. Interfacial tension is the measure of free energy per unit area of interface between two liquid phases and it has the potential to characterize changes that occur at the oil/water interfaces.



## Figure 14. Schematics of the structures of film formed at model oil-water interfaces at a) 30 °C and b) 55 °C (Myristic acid molecule $\stackrel{>}{\delta}$ , model oil molecule $\stackrel{>}{\delta}$ ).

The oil-water interfacial tension results for different concentrations of myristic acid at both 30 and 55 °C are shown in Figure 15. The oil-water interfacial tension did not change with temperature, which disagrees with the mechanism proposed above. The oil-water interfacial tension values and correlated corrosion rate results are compared in Figure 16. It was observed that at 30 °C the oil-water interfacial tension decreased by increasing the myristic acid acid concentration which indicates that a more ordered film has formed at the oil-water interface. This film adsorbs on the specimen surface during intermittent wetting cycles and provides a superior corrosion mitigation effect as proven by corrosion rate results. The same behavior was observed at 55 °C in terms of oil-water interfacial tension, however, the corrosion rate results showed that the mitigation effect has decreased with temperature. It seems that the adsorbance and persistency of the layer formed at the oil-water interface to the steel surface has decreased. This implies that interfacial tension values cannot be correlated to corrosion rate results at elevated temperatures.



Figure 15. Oil-water interfacial tensions for 0, 0.1, 1 wt.% myristic acid concentration at pH 4.0 and 30, 55 °C.



Figure 16. Oil-water interfacial tensions and correlated corrosion rates for 0, 0.1, 1 wt.% myristic acid concentration at pH 4.0 and 30, 55 °C.

Effect of elevated temperatures on oil-in-water contact angle

Another possible mechanism is proposed based on the structure of the film formed at the steel-oil interface to explain the effect of elevated temperatures on corrosion behavior of steel. A simple schematic of the structures related to the film formed at these two interfaces is shown in

Figure **17**. Since corrosion rate results showed that the mitigation effect decreases with temperature, it can be postulated that the steel surface becomes more hydrophilic at higher temperatures. Consequently, less myristic acid molecules adsorb on the surface and a less ordered layer forms at the surface.



### Figure 17. Schematics of the structures of film formed at steel-oil interfaces at a) 30 °C and b) 55 °C (Myristic acid molecule $\stackrel{>}{\downarrow}$ , model oil molecule $\stackrel{>}{\downarrow}$ ).

In order to investigate the proposed mechanism, the oil-in-water contact angles were measured. The oilin-water contact angle results for different concentrations of myristic acid at 30 and 55 °C are shown in Figure 18. The contact angle results demonstrated that the affinity of the steel surface changes toward less hydrophilicity by increasing the myristic acid concentration, however, the effect of concentration is more pronounced at 30 °C.



Figure 18. Typical images of model oil droplets containing 0, 0.1, 0.5, 1 wt.% myristic acid on the steel surface in water at: a) 30 °C and b) 55 °C.

The oil-in-water contact angle values and correlated corrosion rate results are compared in Figure 19**Error! Reference source not found.** It was observed that the mesaured contact angle value for 1wt.% myristic acid concentation is lower at 55 °C as compared to 30 °C. This means that the surface is more hydrophilic at 55 °C which results in less adsorption and persistency of the oil layer at the steel



surface and consequently less of a corrosion mitigation effect. The obtained corrosion rate results agree with this mechanism. Therefore, it could be concluded that the effect of temperature on surface wettability is one the parameters that explains the effect of elevated temperature on corrosion mitigation conferred by the oil phase during oil-water intermittent wetting conditions.

#### CONCLUSIONS

- The obtained results demonstrated that the corrosion mitigation effect of the oil phase (model oil containing myristic acid) on CO<sub>2</sub> corrosion decreases with temperature after intermittent wetting.
- For model oil containing myristic acid, the interfacial tension value does not change with temperature (30, 55 °C), and cannot be correlated to the corrosion rate.
- In the presence of model oil containing myristic acid, the surface becomes more hydrophilic at 55 °C, which diminishes the adsorption and persistency of the oil layer and decreases the corrosion mitigation effect.

The effect of temperature on interfacial tension and contact angle may depend on the type of the oil and surface-active compound.

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