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Effect of Acridine and Myristic Acid on CO_2 Corrosion During Intermittent Oil/Water Wetting

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ABSTRACT

The effect of a paraffinic model oil containing select surface-active compounds (myristic acid and acridine) on CO_2 corrosion with and without intermittent wetting has been studied. Observations have shown that the presence of myristic acid in the oil phase has no effect on corrosion behavior due to its lack of partitioning in the water phase. However, after direct contact between the oil phase containing myristic acid and the metal surface, there was a significant decrease in the corrosion rate. This phenomenon gradually diminished at pH 4.0 but was more persistent at pH 6.5. The presence of acridine in the oil phase was shown to have a strong inhibitive effect at pH 4.0, even during the partitioning step. However, there was no inhibitive effect conferred by the presence of acridine on the corrosion rate at pH 6.5. An experimental methodology was developed that facilitated improved simulation of the effect of intermittent oil/water wetting on CO_2 corrosion. The electrochemical current response during the oil/water intermittent wetting cycles showed that persistency of model oil (without surface active compounds) on the mild steel surface is only a matter of seconds. Corrosion rate measurements showed that the presence of myristic acid renders the oil layer more persistent after intermittent wetting as compared to one-time direct contact.

Key words: CO_2 corrosion, carbon steel, oil/water intermittent wetting, oil film, naturally occurring surface-active compounds

INTRODUCTION

Pipelines are the most effective way to transport oil and natural gas, particularly for their bulk transmission over long distances[1,2]. Corrosion in oil and gas pipelines occurs because of the presence of dissolved corrosive gases, such as CO_2 and/or H_2S, in reservoir derived brine, and contact between this brine and the steel surface. The oil phase by itself does not cause corrosion and can even inhibit corrosion [3,4,5,6]. In fact, many surface-active compounds naturally exist in crude oils [7]. Crude oils can be defined as naturally occurring liquid mixture 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 [8,9]. 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, thereby altering the wetting and corrosion properties of steel surfaces [10]. Therefore, if the water phase in a pipeline was entirely entrained by the hydrocarbon phase, instead of flowing at the bottom, no corrosion problems are expected to occur. Therefore, it is important to determine whether the pipe internal surface is wetted by oil or water depending on the fluid properties and the operating flow rates [10]. This knowledge can help decrease economic costs and mitigate the potential for adverse environmental impacts caused by leakage from corroding tubular steels.

In oil-water flow through a horizontal pipeline at low rates, the water phase flows as a separate layer at the bottom of the pipe. This corresponds to a stratified flow regime because gravitational force dominates over turbulent force. By increasing the flow rate inside the pipeline, the turbulent energy increases and, as a result, the water phase becomes gradually entrained as droplets in the oil phase[11]. An example of this can be seen through research conducted with 10% and 1% water cuts as shown in Figure 1: in the image on the left, a separate layer of water on the bottom half surface of the pipe is observed, and in the image on the right the water phase is mostly observed in the form of droplets. Therefore, water may not always wet the bottom surface of the pipe completely, leading to very different corrosion rates depending on the operating scenarios. The measurement of iron count in this example showed the corrosion rate was about 10 times higher when the water content was increased from 1 to 10% while other conditions were kept constant[12].

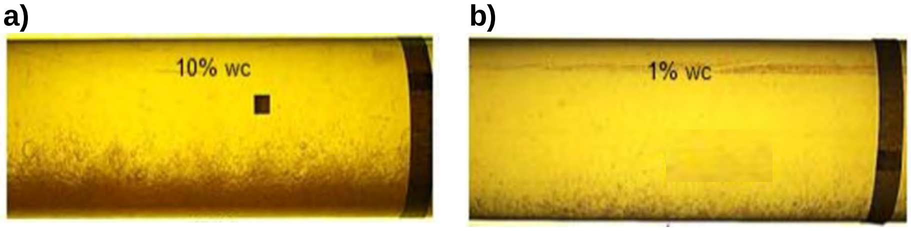

Figure 1. Images of horizontal flow patterns of oil/water flow (LVT-200 & water) at mixture liquid velocity of 1.0 m/s for: a) 10% water cut; and b) 1% water cut[12].

The water phase is typically entrained by the hydrocarbon phase when the water content is low, as a result the oil phase wets the internal surface of the pipe, and there would be no corrosion problem. By increasing the water content, water breakout may occur, consequently, water may more easily wet the surface of the pipe, and corrosion is possible[13,14,15]. Considering that production flow rates can vary significantly during the life of pipeline systems, 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”[16]. Flow rate and water cut can affect the degree of intermittent wetting[15]. This phenomenon can influence the corrosion mechanism of the metal surface, which may differ from those commonly accepted for aqueous corrosion (*i.e.*, full water wetting) [6,17,18]. Consequently, wetting condition of the metal surface is a key parameter to predict corrosion behavior [19,20,21,22], and it is important to know the relation between wetting of the surface and corrosion processes [23,24,25].

EXPERIMENTAL PROCEDURE

Materials and Chemicals

The metal specimens were carbon steel X65 specimens used for electrochemical measurements. The composition of crude oils is complicated and contains many different chemical compounds. Therefore, model oils containing select dissolved species were used to simulate crude oil behaviors in order to isolate their influence. In this study, LVT-200 model oil, which is a hydrotreated light distillate petroleum fraction[26], was used with and without the addition of 0.1 wt.% of two different surface-active compounds, myristic acid and acridine, in the conducted experiments. However, acridine is not soluble in LVT-200 due to the oil's low aromaticity. Therefore, acridine was dissolved in a 60:40 weight ratio of LVT-200 and Aromatic-200. Aromatic-200 model oil contains a complex mixture of aromatic hydrocarbons which are mostly derivatives of naphthalene[27]. Myristic acid and acridine are representative of oxygen and nitrogen containing compounds, respectively, which naturally exist in crude oils. Further, the surfactants concentration used in this study are in the common range of naturally occurring compounds concentration in crude oils. The molecular structures of these surface-active species are shown in Figure 2. Note the amphiphilic character of myristic acid, and the aromaticity of acridine. Myristic acid has a low solubility in water, however acridine is soluble in water at low pH values[25].

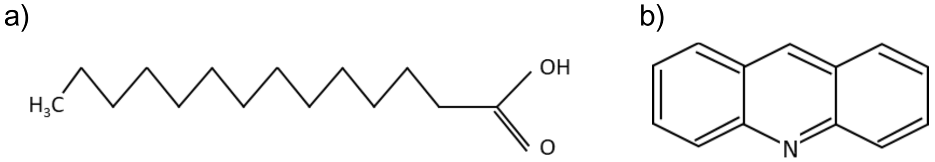

Figure 2. Molecular structures of; a) myristic acid; and b) acridine.

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 for 1 hour by sparging with CO_2 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 test corrosion rate to ensure no contamination occurred from the previous test. Linear polarization resistance (LPR) measurements were taken using a Gamry potentiostat. To minimize the noise in electrochemical measurements caused by CO_2 sparging, the sparge tube was retracted into the headspace during data acquisition. The solution was sparged with CO_2 throughout the test to prevent air (oxygen) ingress and to saturate the test solution with CO_2. The pH was adjusted by adding deoxygenated hydrochloric acid or sodium bicarbonate solution during each experiment. The experiments were performed using two different procedures: one-time direct contact or intermittent wetting.

I. <u>One-time direct contact:</u> The main glass cell was connected to a separate side glass cell used to change the level of the water phase in the main glass cell, enabling immersion of the specimen in oil and water, alternately. A schematic of this setup is shown in Figure 3. In this method, the experiments were performed in four steps:
    1. Pre-corrosion: The polished specimen was immersed in the water phase.
    2. Partitioning: 0.3 liters of an oil layer containing a surface-active compound was added to the top of the water phase. The polished specimen remained immersed in the water phase and rotated for one hour at 1000 rpm. Corrosion rate by LPR was measured every 20 minutes.
    3. Direct inhibition by oil: The water was drained from the side port of the glass cell until the polished specimen was immersed into the oil phase and rotation continued for one hour at 1000 rpm.
    4. Persistency: After immersion in the oil layer, fresh brine, prepared and sparged in the side glass cell, was gravity fed into the main glass cell so that the specimen was returned to the water phase. Corrosion rate by LPR was measured every 20 minutes until reaching a stable value.

    A schematic of the procedure for these experiments is shown in Figure 4. The test matrix used to study the effect of direct contact with oil on corrosion rate is presented in Table 1.

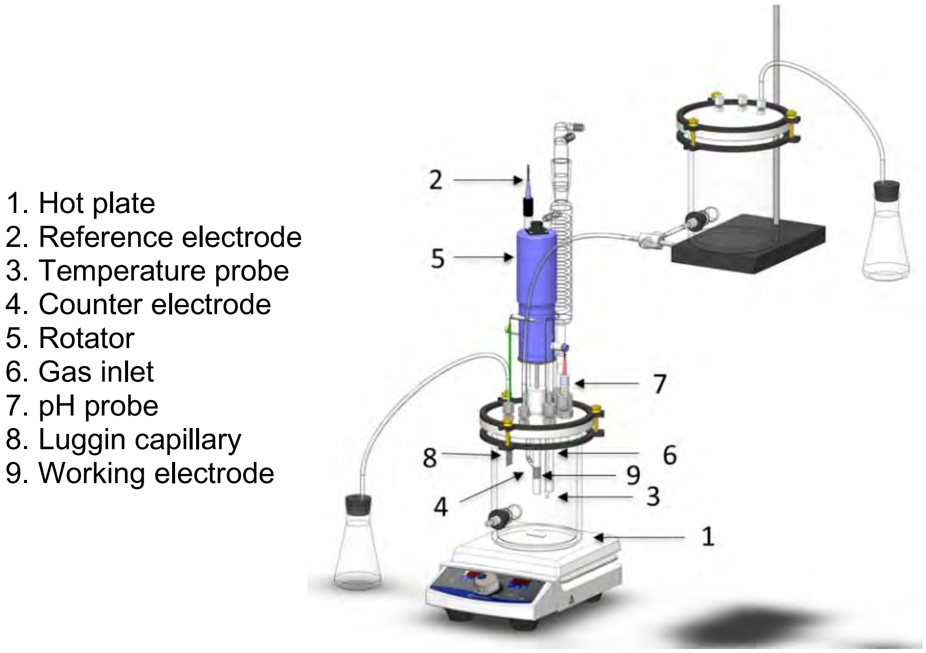

Figure 3. Schematic of main and side glass cells. The side glass cell is located on a shelf above the main cell.

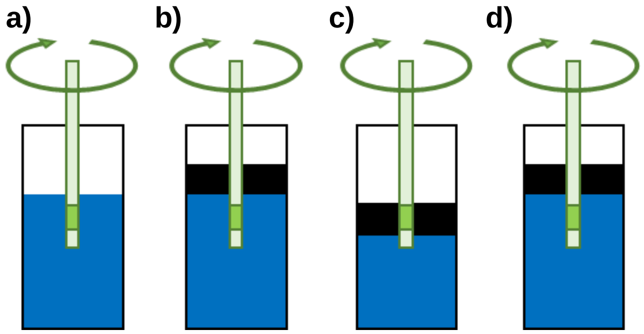

Figure 4. Procedure of one-time direct contact experiments: a) pre-corrosion; b) partitioning; c) direct inhibition by oil; and d) persistency.

Table 1. Test matrix to study the effect of one-time direct contact with oil on corrosion rate.

Parameter	Conditions
Material	X65
Solution	1 wt.% NaCl/model oil (15% of total liquid)
Model oil	LVT-200 (for myristic acid), 60:40 weight ratio of LVT-200 and Aromatic-200 (for acridine)
Surface-active compounds	Myristic acid, acridine (0.1 wt.% of oil phase)
Wetting regime	Direct contact with oil
Temperature	30°C
CO_2 partial pressure	0.96
pH	4.0, 6.5
Experimental equipment	RCE, Glass cell
Electrochemical measurements	OCP, LPR, EIS, potentiodynamic

II. Intermittent wetting: 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 5. Experiments were performed therein in four steps as well, with the third step being different from the previous method. In this method, the "inhibition by oil" step consisted of alternating between oil wetting and water wetting instead of direct continuous contact with oil in order to facilitate improved simulation of 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 surrounds 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.

The wetting periods were initially repeated every 30 seconds for at least one hour. Since LPR measurements could not be performed during the intermittent wetting cycles (a cycle duration is too short to allow collection of LPR data), a different method had to be used to assess the corrosion response. To do so, the electrode potential was held at an arbitrary anodic potential of +60 mV *vs.* OCP, and the current response was collected. It is understood that the measured current does not correspond to the natural corrosion rate of the specimen, however, it can give indication on whether or not corrosion occurs.

Most of the experiments were performed considering wetting periods that were repeated every 60 seconds for one hour. 60 seconds cycles proved to be more practical than 30 seconds and yielded no significant difference on the results. For this sets, LPR was performed after the cyclic wettings. A schematic picture of the procedure for these experiments is shown in Figure 6. The test matrix used to study the effect of intermittent contact with oil on corrosion rate is presented in Table 2.

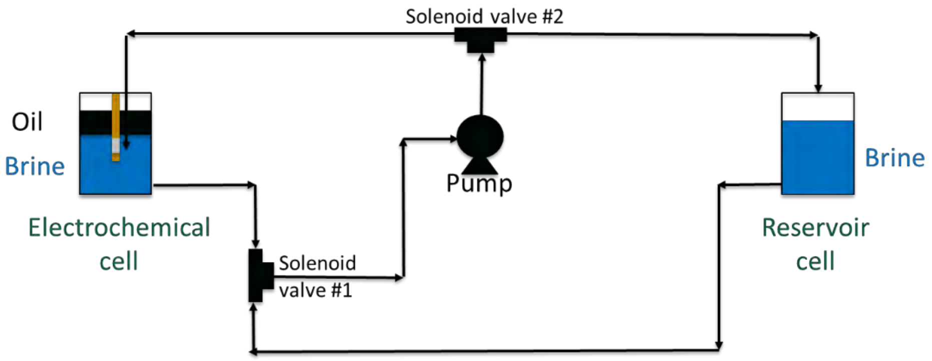

Figure 5. A simple schematic of the intermittent wetting setup.

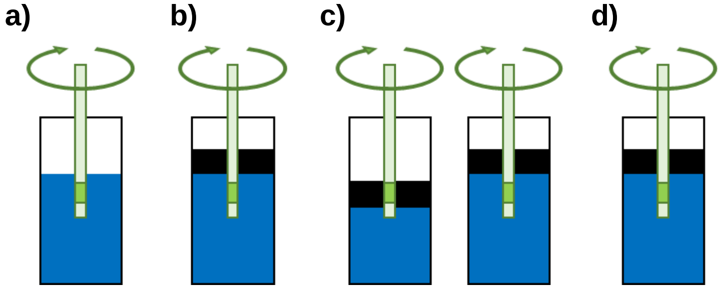

Figure 6. Procedure of intermittent wetting experiments: a) pre-corrosion; b) partitioning; c) oil/water intermittent wetting; and d) persistency.

Table 2. Test matrix to study the effect of intermittent wetting on corrosion rate.

Parameter	Conditions
Material	X65
Solution	1 wt.% NaCl/LVT-200 (15% of total liquid)
Surface-active compounds	Myristic acid
Wetting regime	Intermittent wetting
Temperature	30°C
CO_2 partial pressure	0.96
pH	4.0
Rotational speed	Stationary, 1000 rpm
Intermittent wetting period	30, 60 seconds
Experimental equipment	RCE, Glass cell
Electrochemical measurements	OCP, LPR, EIS, Potentiostatic

RESULTS

Effect of one-time direct contact with oil on corrosion rates

Effect of myristic acid

The results of baseline experiments in the absence of an oil phase and one-time direct contact experiments in the presence of LVT-200 containing 0.1 wt.% myristic acid at pH 4.0 are shown in terms of corrosion rate *vs.* time in Figure 7. A stable uniform corrosion rate of around 4 mm/year was observed in the baseline experiment. In the repeated one-time direct contact experiments the corrosion rate did not change in the partitioning step, however, there was a major decrease after the direct contact with the hydrocarbon phase which gradually diminished[28]. The results of baseline experiments in the absence of an oil phase and one-time direct contact experiments in the presence of LVT-200 containing 0.1 wt.% myristic acid at pH 6.5 are shown in Figure 8. A stable uniform corrosion rate of around 1.5 mm/year was observed in the baseline experiment. In the one-time direct contact experiment, there was again no effect in the partitioning step, a significant decrease in the corrosion rate after the direct inhibition step, and a stable value in the persistency step.

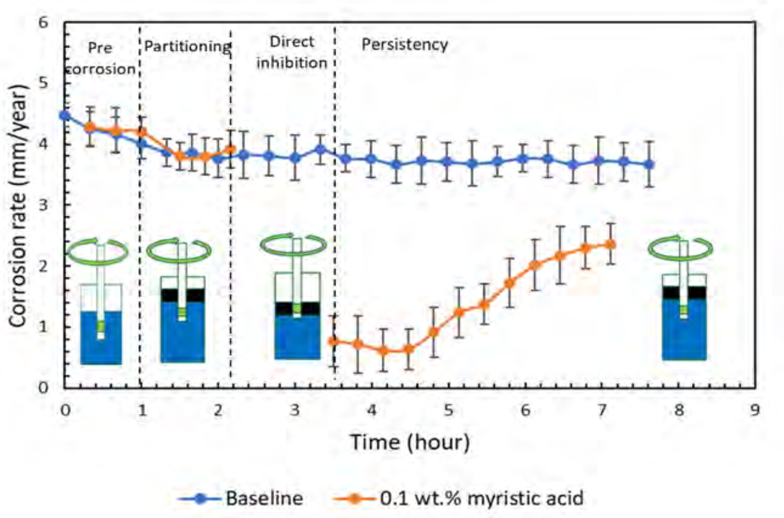

Figure 7. Corrosion rate in the absence and presence of LVT-200 containing 0.1 wt.% myristic acid at pH 4.0, 30°C, 0.96 bar CO_2 and 1000 rpm.

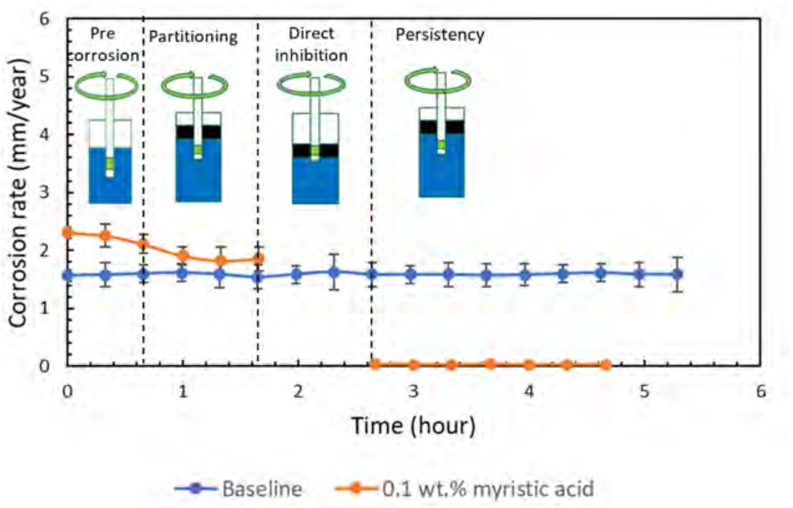

Figure 8. Corrosion rate in the absence and presence of LVT-200 containing 0.1 wt.% myristic acid at pH 6.5, 30°C, 0.96 bar CO_2 and 1000 rpm.

The results are dependent on pH, which is a property of the aqueous phase. Myristic acid has a very low solubility in water and no effect on corrosion was observed during the partitioning tests at pH 4.0 or 6.5. Consequently, it can be postulated that the pH of the water phase affects the nature of the species present at the oil/water interface, which is in turn related to the observed corrosion behavior.

Looking first at the effect of pH, the pK_a value for myristic acid is 4.9, which means at pH values higher than 4.9 it increasingly exists as myristate anions.

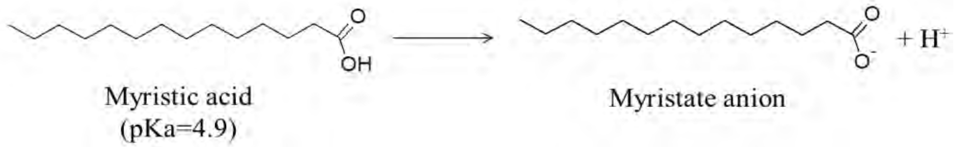

Figure 9. Dissociation of myristic acid

In order to compare the results at different pH values, the Henderson-Hasselbalch equation is applied as follows[29]:

$$pH = pKa + \log\left(\frac{[A^-]}{[HA]}\right) \qquad \text{Equation 1}$$

According to Equation *1*, at pH 4.0 the ratio of myristate to myristic acid is equal to:

$$\frac{[Myristate\ anion]}{[Myristic\ acid]} = 0.13$$

And at pH 6.5 this ratio is equal to:

$$\frac{[Myristate\ anion]}{[Myristic\ acid]} = 39.8$$

Assuming that these comments are applicable to the oil/water interface, it can be hypothesized that the interface is mainly made of myristic acid at pH 4.0, while it is mostly made of myristate ions at pH 6.5. In addition, it can also be postulated that the layer adsorbed at the oil water interface at pH 6.5 is denser and more compact since myristate ions cannot exist in the oil phase (as opposed to myristic acid).

During the wetting cycles, the steel specimen crosses this interface twice (water to oil and oil to water) and it is likely that the structure of the layer at the oil/water interface is replicated on the steel surface, as it will be discussed later on in this paper. Following all these assumptions, the effect of myristic acid on the corrosion rate could be explained by the nature of the oil/water interface:

- At pH4, the adsorbed layer is mostly made of myristic acid. The layer provides initially some protection but is quickly penetrated by water and is not persistent, probably due to the loose organization of molecules within the layer.
- At pH6.5, the adsorbed layer may be made of more densely packed myristate ions, which offers better corrosion protection and better persistency than myristic acid.

Obviously, these comments need further validation.

Effect of acridine:

The results of one-time direct contact experiments in the presence of mixture of LVT-200 and Aromatic-200 containing 0.1 wt.% acridine at pH 4.0 are shown in terms of corrosion rate *vs.* time in Figure 10. In this experiment, the corrosion rate started to decrease in the partitioning step until it reached a stable value, and it did not change after direct contact with the hydrocarbon phase. The results of one-time direct contact experiments in the presence of mixture of LVT-200 and Aromatic-200 containing 0.1 wt.% acridine at pH 6.5 are also shown in Figure 11. In this experiment, there was no effect on the corrosion rate either in the partitioning step or even after the direct inhibition step. The results of the potentiodynamic sweeps taken at the end of each experiment agree with the corrosion rate measurements. The potentiodynamic sweep at pH 4.0 is shown in Figure 12.a. Both cathodic and anodic reactions were retarded by the presence of acridine in the oil phase. At pH 6.5, however, both cathodic and anodic reactions remained unaffected, as shown in Figure 12.b.

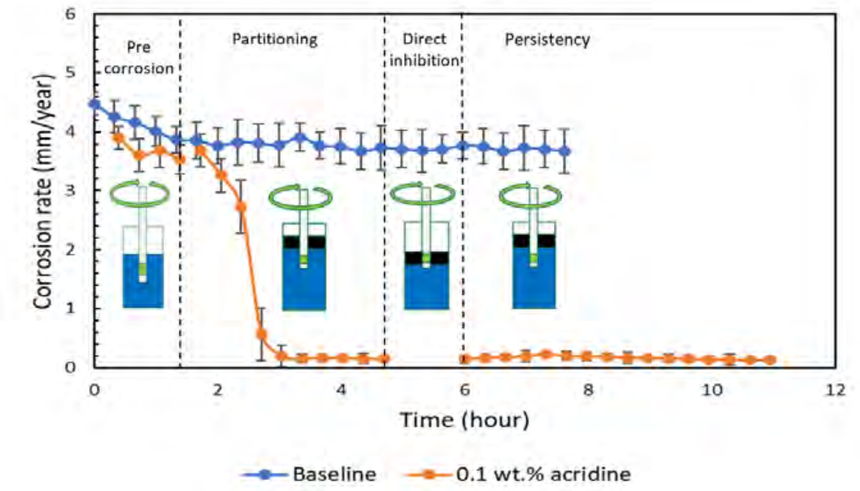

Figure 10. Corrosion rate in the absence and presence of mixture of LVT-200 and Aromatic-200 containing 0.1 wt.% acridine at pH 4.0, 30°C, 0.96 bar CO_2 and 1000 rpm.

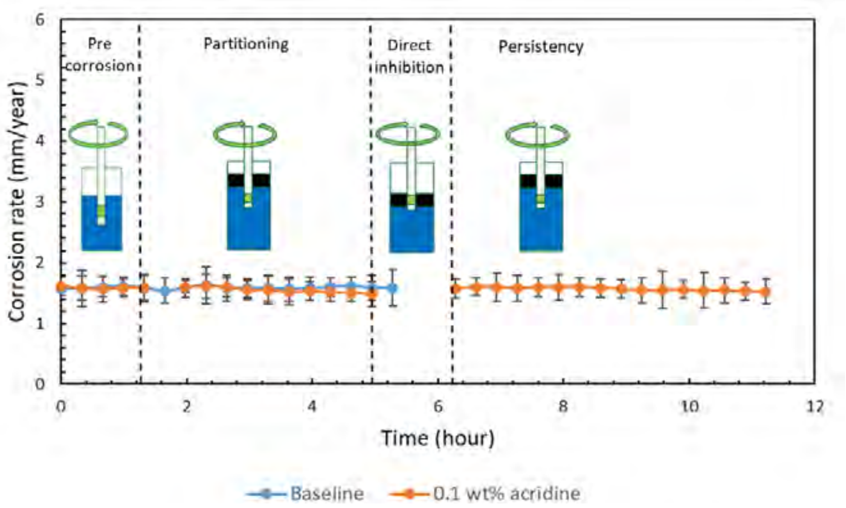

Figure 11. Corrosion rate in the absence and presence of mixture of LVT-200 and Aromatic-200 containing 0.1 wt.% acridine at pH 6.5, 30°C, 0.96 bar CO_2 and 1000 rpm.

a) **b)**

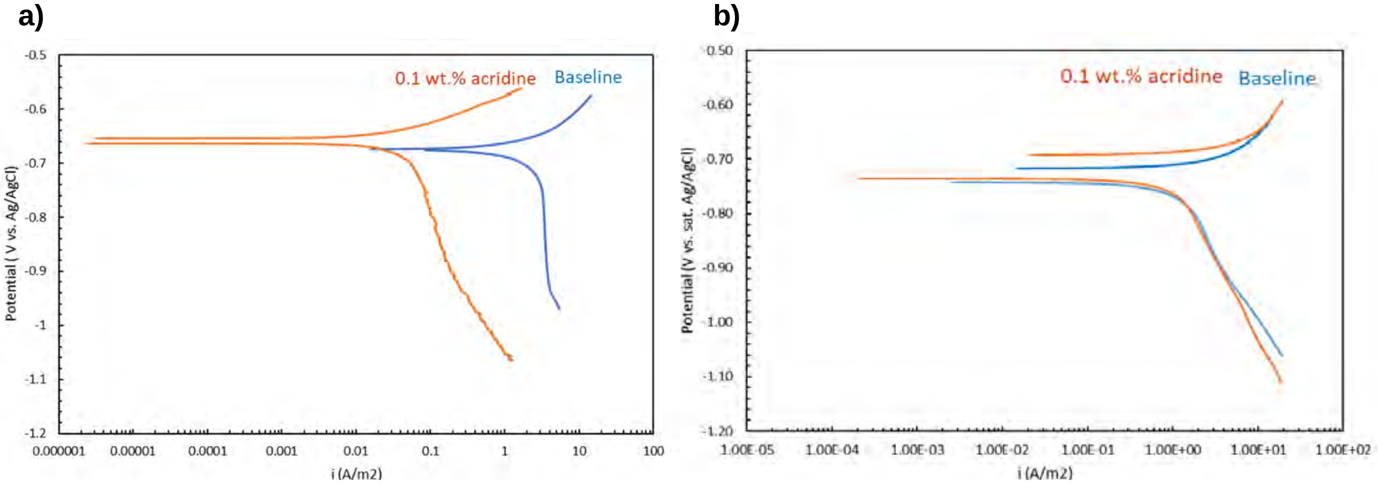

Figure 12. Potentiodynamic sweeps for experiments conducted in the presence of mixture of LVT-200 and Aromatic-200 containing 0.1 wt.% acridine at: a) pH 4.0; and b) pH 6.5.

The results are once again pH dependent. However, acridine is much more soluble in water, especially at pH 4, as the results of the partitioning step show. The pK_a value for the acridinium cation is 5.6, which means at pH values lower than 5.6, acridine increasingly protonates to form acridinium cations. According to Equation 1, the ratios of acridine to acridinium at pH 4.0 and pH 6.5 can be readily calculated and used to explain the corrosion rate results. A possible explanation for the inhibitive effect of acridine at different pH values could be that acridinium ions can partition through the water phase and adsorb at the steel surface. The concentration of acridinium is high at pH 4.0, the acridine:acridinium ratio is 0.0251. Consequently, it can inhibit corrosion and reduce the corrosion rate. At pH 6.5, however, the acridine:acridinium ratio is 7.94 and, due to the higher concentration of acridine, which is not soluble in water and less surface-active than acridinium, there is almost no effect on the corrosion rate.

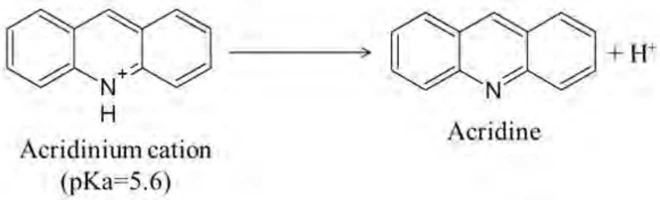

Figure 13. Dissociation of acridinium

It is also important to mention that the nature of the oil/water interface played a much smaller role in the corrosion behavior, compared to the case of myristic acid. Acridine does adsorb at the oil/water interface but does not orient in an organized fashion, the way that amphiphilic myristic acid does. It can be postulated that the structure of the layer absorbed on the steel surface is similar to the interface, yielding little effect in terms of corrosion protection.

It was observed that acridine can partition from the oil phase to the water phase, adsorb on the specimen surface, and provide corrosion mitigation at pH 4.0. Therefore, performing cyclic oil-water wetting would not change its inhibitive behavior on CO_2 corrosion. On the other hand, myristic acid did not partition from the oil phase to the water phase. Therefore, performing cyclic oil-water wetting experiments would test the persistency of the oil film containing myristic acid on the specimen surface during the water wetting cycling. Moreover, the corrosion mitigation effect provided at pH 6.5 in the presence of myristic acid is relatively high and persistent. Consequently, only myristic acid was used, at pH4.0, in the next series of experiments: alternative oil/water wetting experiments.

Effect of oil/water intermittent wetting on corrosion rates

Effect of LVT-200:

An anodic potential of +60mV *vs.* OCP was applied to directly measure the electrochemical current response of the specimen to oil/water intermittent wetting and the transient current response, in the absence of the oil phase, is shown in terms of current *vs.* time in Figure 14. A stable uniform current response was observed under full water wetting condition, which indicates a steady active dissolution of the specimen surface. An anodic potential of +60mV *vs.* OCP was also applied under full oil wetting condition and the transient current response is shown in terms of current *vs.* time in **Error! Reference source not found.**Figure 14. A constant zero current response was observed under full oil wetting conditions. As shown in Figure 14**Error! Reference source not found.** and Figure 15, the electrode was immersed in water, then in oil, alternating every 30 seconds. The anodic current response in these experiments changed periodically between a stable value and zero which is in agreement with the literature[30]. It was observed that the current response decreased gradually as the oil replaced the water on the specimen surface. However, it increased rapidly to the initial value as the water replaced the oil on the specimen surface. As seen from**Error! Reference source not found.**, the influence of LVT-200 model oil with no additives shows no persistency on the specimen surface as the measured current increases and decreases similar to the amount of specimen surface area remaining in the brine phase. It is speculated that this very fast response is an indication that a thin water layer always remains on the specimen surface.

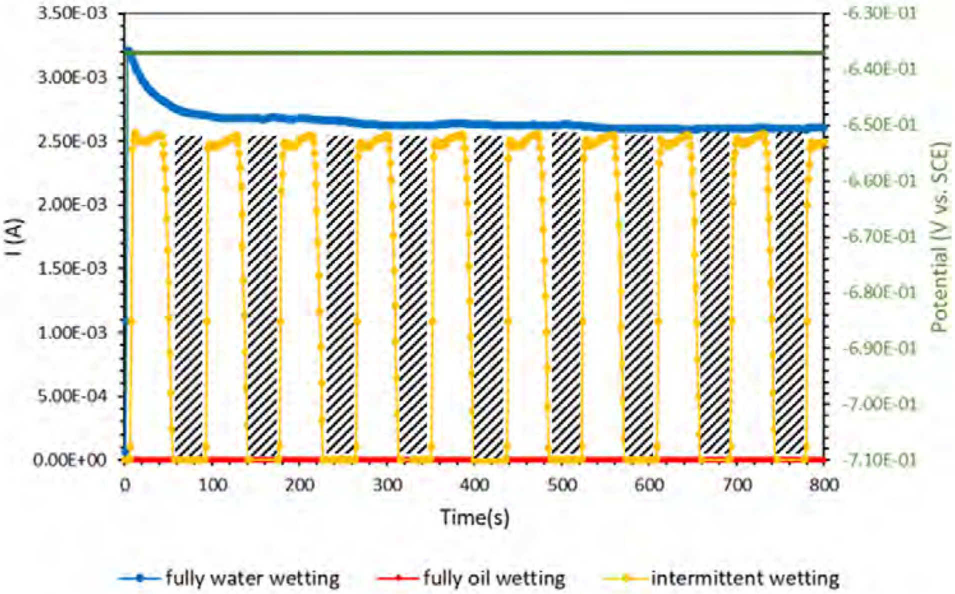

Figure 14. Transient current-time response under fully water wetting, fully oil wetting and intermittent wetting conditions using LVT-200 model oil, blue, red, and yellow curves are currents (I (A)), ▨ oil wetting part of the intermittent wetting cycle.

Effect of LVT-200 containing myristic acid:

An anodic potential of +60mV *vs.* OCP was applied and the transient current response under intermittent wetting condition in the presence of LVT-200 containing 0.1 wt.% myristic acid at pH 4.0 is shown in terms of anodic current *vs.* time in Figure 15. A lower current response was measured in water wet periods, as compared to the current response for water wet periods of intermittent wetting with model oil alone. It can be postulated that model oil containing myristic acid developed a persistent layer on the specimen surface which was not observed for the model oil alone. The pattern of changes in current during upward and downward movement of the oil layer was also observed to be different. Unlike the experiment with LVT-200, it cannot be assumed that a thin layer of water remains on the surface.

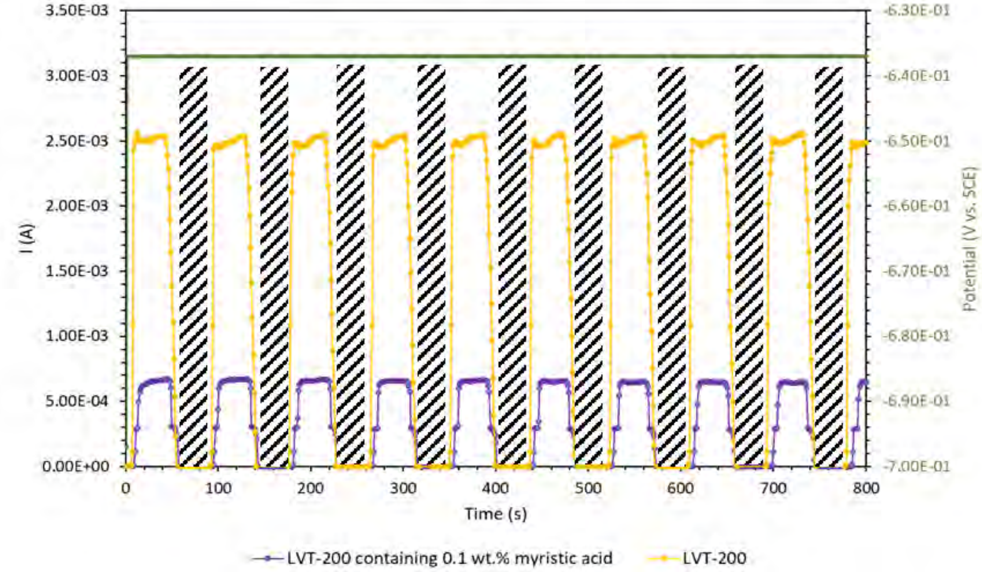

Figure 15. Transient current-time response under intermittent wetting conditions in the presence of LVT-200 containing 0.1 wt.% myristic acid.

The current-time results agreed with the observed surface wettability. The specimen surface wettability in the presence of LVT-200 alone and LVT-200 containing myristic acid are shown in Figure 16. In the presence of LVT-200 alone, the specimen surface was observed to be hydrophilic, while in the presence of 0.1 wt.% myristic acid dissolved in LVT-200, the specimen surface became hydrophobic.

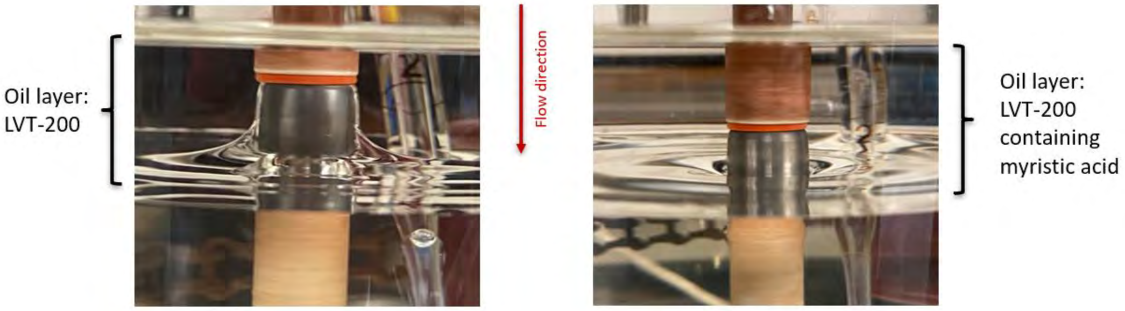

Figure 16. Surface wettability state in the presence of LVT-200 alone and LVT-200 containing 0.1 wt.% myristic acid.

The results for corrosion rate experiments under intermittent wetting conditions in the presence of LVT-200 containing 0.1 wt.% myristic acid are shown in Figure 17. In this experiment, the specimen was periodically immersed in each phase for 60 seconds 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 results for corrosion rate experiments under one hour fully oil wetting condition in the presence of LVT-200 containing 0.1 wt.% myristic acid is also shown in Figure 17. In this experiment, the specimen was continuously immersed in the oil phase for one hour. The corrosion rate did not change in the partitioning step, while there was a major decrease after the direct contact with the hydrocarbon phase which was gradually diminishing. Different corrosion behaviors observed for these two experimental methods could be related to the structure of film formed on the specimen surface. Amphiphilic myristic acid molecules orient themselves at the oil/water interface and form a monolayer. When the specimen crosses this interface, the monolayer is likely to adsorb to the specimen surface. During intermittent wetting experiments the specimen crosses this interface 60 times,

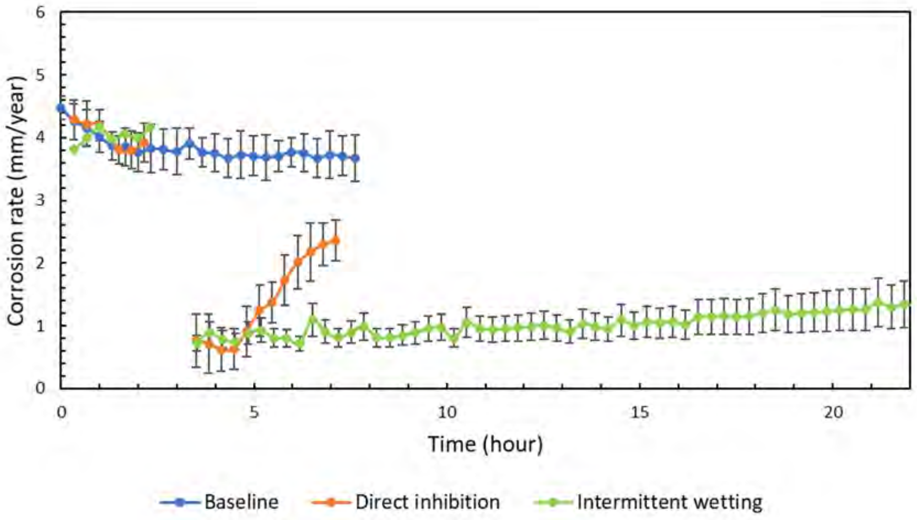

Figure 17. Corrosion rate in the presence of LVT-200 containing 0.1 wt.% myristic acid at pH 4.0, 30°C, 0.96 bar CO_2 and 1000 rpm for one-time direct contact with oil (orange) and one-hour intermittent wetting (green).

however for one-time direct contact experiments the specimen crosses the interface only twice. A possible mechanism to explain the stable effect of myristic acid on corrosion rate after intermittent wetting cycles is that increasing the number of contact times between the specimen surface and the oil/water interface promotes the development of a more persistent layer (a multilayer), as compared to a one-time direct contact.

CONCLUSIONS

- The addition of myristic acid and acridine to a model oil decreased the CO_2 corrosion rate through adsorbing on the steel surface and changing the wettability of the steel surface from hydrophilic to hydrophobic.
- Varying pH of the aqueous phase above or below the pK_a of myristic acid and acridinium will alter their properties through ionization processes. This will result in significant changes in corrosion inhibition behavior of these surface-active compounds.
- The presence of myristic acid in the oil phase has no effect on corrosion behavior in the partitioning step. After the direct inhibition step, however, there was a significant decrease in the corrosion rate, which gradually diminished at pH 4.0 but was more persistent at pH 6.5.
- The presence of acridine in the oil phase has a strong inhibitive effect at pH 4.0, even during the partitioning step of testing. While there was no effect of the presence of acridine on the corrosion rate at pH 6.5.
- Persistency of model oil with no additives on the mild steel surface was in the order of seconds. It is speculated that water never leaves the specimen surface and may always exist as a thin layer (an initially water wet metal surface stayed water wet).
- An LVT-200 model oil layer containing myristic acid replaced the water on the specimen surface and formed a much more persistent layer as compared to LVT-200 model oil alone.
- The presence of myristic acid renders the oil layer more persistent after intermittent wetting as compared to one-time direct contact.

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