

Influence of Experimental Injection Method on the Inhibition Efficiency of Volatile Corrosion Inhibitors for Top-of-the Line Corrosion Mitigation

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ABSTRACT

The extent of Top-of-the-Line Corrosion (TLC) can be reduced using volatile corrosion inhibitors (VCIs). The selection of the correct VCIs depends of course on the operating conditions ($p\text{CO}_2$, $p\text{H}_2\text{S}$, temperature, hydrocarbon co-condensation, organic acid concentration) and on the intrinsic properties of the inhibitor itself (water solubility, volatility, active components). Yet, the method used to inject the VCIs inside the producing pipeline could also be of practical importance, especially considering the complex environment of a multi-component condensing system. A concern among the related industries is the lack of a methodology that would allow for conducting experiments mimicking the field conditions and provide a guideline for extrapolating the experimental results to a larger scale. In this study, a methodology was developed to investigate the effect of injection methods on the inhibition efficiency of VCIs (model compounds decanethiol and hexanethiol). For this purpose, a system of two connected glass cells was designed enabling the injection of the VCIs into the system either through the liquid or directly into the gas phase. The experiments were conducted in the presence of a condensable hydrocarbon (n-Heptane) to investigate the partitioning effect. The results show that injection of the inhibitor through the gas or liquid phase has no influence on its inhibition efficiency, as long as the VCI is sufficiently volatile. Moreover, regardless of the injection method, the hydrocarbon phase interferes with the inhibition by decanethiol while it has no influence on hexanethiol. For a larger scale, it can be concluded that any length of pipe can be protected by an inhibitor injected through the liquid or gas phase if the inhibitor is an effective VCI.

Key words: Keywords: Top of the Line Corrosion, Carbon Steel, Volatile Corrosion Inhibitor, Corrosion Inhibition, Hydrocarbon, Inhibition Efficiency.

INTRODUCTION

In oil and gas industry, during the transportation of wet gas with a stratified flow regime, the temperature difference between the fluid inside the pipelines and the surrounding environment leads to condensation of water on the upper internal surface and causes metal degradation. This phenomenon is known as Top-of-the-Line Corrosion or TLC.

The condensing phases can consist of not only water but also condensable hydrocarbons. While the condensed hydrocarbon phases will not cause any corrosion to the pipeline, the condensed water phase is corrosive due to the presence of dissolved acid gases (CO_2 and H_2S) and organic acids (acetic acid [CH_3COOH] and formic acid [HCOOH]) in it. ¹ The mechanism of TLC is not different from that of $\text{CO}_2/\text{H}_2\text{S}$ corrosion in the bulk liquid phase at the bottom of the pipeline.² However, TLC inhibition/mitigation is generally not possible by using the conventional $\text{CO}_2/\text{H}_2\text{S}$ inhibitors. This is mainly because the inhibitors injected into the liquid phase in the pipeline will not reach its upper surface, where the TLC occurs and therefore, their application remains limited to the surfaces wetted by the bulk water phase. ³

Among the practical TLC inhibition/mitigation strategies, batch corrosion inhibition ⁴⁻⁷ has been studied and applied in field conditions in recent years. In batch inhibition treatment, a plug of fluid containing high content of inhibitor is circulated through the pipeline between two inspection gauges (the so-called pigs). A similar method involves the use of specially designed spraying device. ⁸ The batch inhibition treatment requires shutdown of the operation which leads to a massive production loss, moreover, not all the pipelines in service are compatible with the pigging technology. ⁹ In fact, the applicability of pigging technique is restricted by piping design elements such as bends, miters, inline restrictions, changes in diameters, etc. ¹⁰ Success of the batch treatment mainly depends on the inhibitor persistency which requires stronger bond of adsorption between the inhibitor and the metal surface. ¹¹

Over the years, other techniques have been developed for the transport of inhibitor along the pipeline. One of these techniques involves the use of a foam slug as the inhibitor carrier matrix in the pipeline. ¹² ¹³ This method requires detailed chemical fitness analysis between foaming agents and the inhibitors which increases the effort and limits the trial of new inhibitors.

Due to practical and economical restrictions of above-mentioned techniques, operators are compelled to look for inhibitors, such as volatile corrosion inhibitors (VCIs) ^{3, 14-17} that can be injected continuously in the pipeline. However, injection of inhibitors in the pipe flow with stratified pattern in case of TLC is challenging. The transport of a VCI, typically injected in the bulk liquid phase at the inlet of the pipeline to the top wall and all along the line strongly depends on the vapor pressure of the selected inhibitor. For the inhibitors with high vapor pressure, a greater partitioning in the vapor phase is expected which leads to a better reach to the top wall. However, other factors, such as partitioning of most of VCIs in the hydrocarbons present over the bulk aqueous phase at the bottom of the line, influence, and in some cases, interfere with the inhibition efficiency.^{3, 16, 18}

No universal method exists for effective inhibitor treatment of pipelines using VCIs. Challenges remain to formulate the most effective inhibitors combination and appropriate application method for TLC of wet gas pipelines. More importantly, experimental methodologies to investigate the effectiveness of the selected injection method in laboratory scale are needed as well.

The corrosion inhibition efficiency of VCIs depends on the molecular structure of the inhibitor, the nature of its interaction with the metal surface, and possibly the environmental conditions of the application.¹⁹ Similarly, published studies on the application of volatile corrosion inhibitors for TLC also

include those on the effect of type of functional groups (such as amines and thiols)^{3, 14} and operating parameters (such as water condensation rate, the acid content, and the presence of hydrocarbon phases)^{16, 18} on the inhibition efficiency.

In recent years, a number of testing methodologies and experimental setups have been used to evaluate VCI efficiency. One of the setups, that has been often utilized to investigate TLC and the inhibition efficiency of model volatile compounds, consists of a glass cell, on a hot plate, in which the specimen is placed in its top lid, exposed to the gas phase. In this setup, in order to increase the water condensation rate the specimen is usually cooled down externally in order to increase the water condensation rate.²⁰ This setup has been proved useful in studying the effect of operating parameters on TLC rate and the inhibition efficiency of VCIs and batch inhibitors. In this setup, the inhibitor is usually injected in the liquid at the bottom of the glass cell and is transported to the top of the line, hence the surface of specimen, via evaporation and through the gas phase. This, to some extent, highlights the effect of the volatility of the inhibitor and the transport of the inhibitor through the gas phase. However, in the field, when the inhibitor is injected in the pipeline, it is expected to be effective, and consequently transported, over long distances through the pipe via the gas phase. The current common glass cell setup cannot study the effect of injection of the inhibitor through the gas phase on the inhibition efficiency. Modifications to this setup are needed to address this.

As mentioned earlier, one of the important challenges associated with TLC mitigation is the interference of hydrocarbons with VCI inhibition. Despite availability of studies on the effect of hydrocarbon on the inhibition efficiency of VCIs such as thiols,^{3, 16, 18} the interference of the hydrocarbon phase with the inhibition efficiency of VCI when injected through the gas phase has never been studied.

The aim of the present study is to modify the available TLC setup to investigate the effect of different injection methods (through the liquid or gas phase) on the inhibition efficiency of two VCI model compounds (decanethiol and hexanethiol) in the presence of a condensable hydrocarbon phase (n-heptane). The corrosion rate is measured using the weight loss method and the corroded surfaces are evaluated using scanning electron microscopy (SEM) and optical profilometry techniques.

EXPERIMENTAL PROCEDURE

Specimens from a 1018 (UNS⁽¹⁾ G10180) steel were used for the current study. The specimens were machined into a cylindrical shaped sample, with a diameter of 3.15 cm and 1.15 cm thickness. One of the faces and the circumference were covered with Xylan® coating, leaving an exposed area of ~7.8 cm². The exposed surface was polished up to #600 grit using silicon carbide abrasive papers. The specimens were then ultrasonically cleaned in isopropanol for 5 minutes, and dried.

The experimental setup was a 4 L glass cell specially designed for TLC experiments. This glass cell was connected to another 2 L glass cell which was filled with deionized water (DI) and was considered a pre-bubbler. Figure 1 depicts the schematic of this 2-glass cell setup. The pre-bubbler (the 2 L glass cell) was filled with 1.7 L of water. Then, 2.5 L of DI water was poured into the main glass cell and the whole system was sparged with CO₂ for at least 2 hours to facilitate deoxygenation and saturate it with carbonic species. In the experiments that contained a hydrocarbon phase (n-heptane), the volume of DI water in the main glass cell was decreased to 1.7 liters and 0.8 liters of heptane was added to the glass cell after a 2-hour period of CO₂ sparging. In this case, sparging was continued for another hour to remove the remaining oxygen in the system. During the deoxygenation process, the solutions in the main glass cell and the pre-bubbler were heated to 75°C (85°C when heptane was present in the main glass cell) and 90°C, respectively, using a hot plate. This solution temperature leads to a gas temperature of 65°C in the main glass cell.

⁽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.

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After the desired solution temperature was reached, the gas-in tube in the main glass cell was raised into the gas phase to avoid any assisted transportation of VCI from the bottom solution to the top surface via aerosol formation. Subsequently, 600 ppm of acetic acid was added to the solution in the main glass cell and 400 ppm of the inhibitor (decanethiol or hexanethiol) was added to the solution either in the main glass cell or in the pre-bubbler, depending on the experiment. After injecting the inhibitor, the specimen was flush mounted to the lid, exposing the polished surface to the wet gas phase. To control the water condensation rate, the specimens were heated to 56 ± 1 °C by circulating water through a heating/cooling system. The temperature of the gas, solution and steel specimen was monitored during the experiment. The duration of each experiment was 3 days.

The test matrix is summarized in Table 1. As described in this table, in addition to the blank test, which represents the experiment in the absence of an inhibitor, two experiments were carried out by injecting the inhibitor in the pre-bubbler solution (with and without a hydrocarbon phase present in the main glass cell). This was done to investigate the transportation of the VCI and its corrosion inhibition when it is injected through the gas phase. Therefore, these experiments are denoted by Inh-G. Two other experiments were conducted with the inhibitor injected in the liquid phase in the main glass cell with and without the hydrocarbon phase as well. These experiments are denoted by Inh-L.

Similar experiments to those done with model compounds (decanethiol and hexanethiol) were executed using a commercial inhibitor from purchased from Arkema[†]. The main active compounds in this inhibitor were amin based. In this case the recommended inhibitor dosage by the manufacturer was 2000 ppm as noted in Table 1.

After each experiment, surface of the specimens was characterized using scanning electron microscopy (SEM, JEOL[‡] JSM-6090). The corrosion products on the specimens were removed using Clarke solution, according to ASTM Standard G1-03²¹. Clean surfaces were investigated for localized corrosion using optical profilometry.

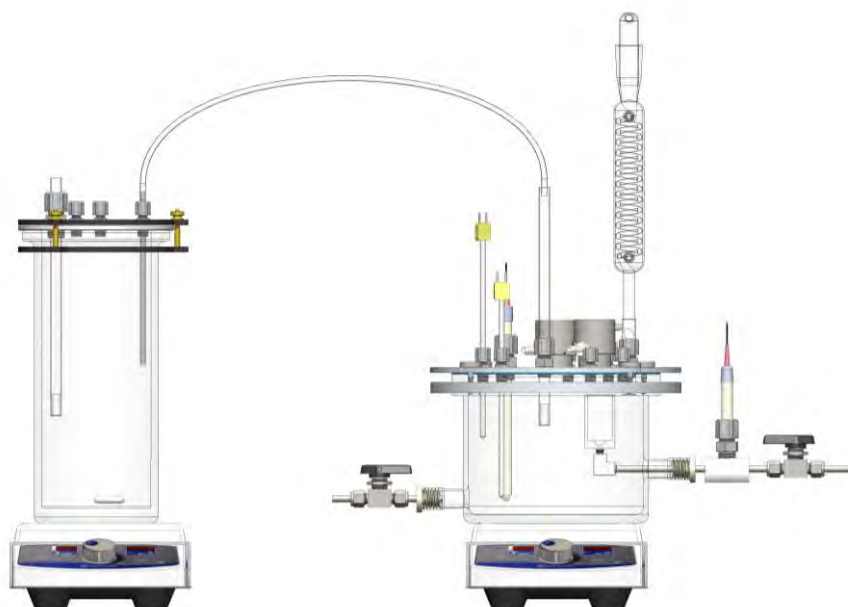


Figure 1: The 2-glass cell setup for TLC experiments.

[†] Trade name.

[‡] Trade name.

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Table 1: Test matrix of glass cell experiments

Test ID*	Water condensation rate (ml/m ² /s)	Injection method	Decanethiol (ppm _v)	Hexanethiol (ppm _v)	Commercial Inhibitor (ppm _v)	Heptane (L)
Blank Test	~0.4	No inhibitor	0	0	0	0
Blank-HWCR	~0.7	No inhibitor	0	0	0	0
Blank Test-Heptane	<0.1	No inhibitor	0	0	0	0.8
Decanethiol (Inh-G)*	~0.4	Injected in gas phase	400	0	0	0
Decanethiol-Heptane (Inh-G)	<0.1	Injected in gas phase	400	0	0	0.8
Decanethiol-Heptane (Inh-L)*	<0.1	Injected in liquid phase	400	0	0	0.8
Decanethiol (Inh-L)	~0.4	Injected in liquid phase	400	0	0	0
Hexanethiol-Heptane (Inh-L)	<0.1	Injected in liquid phase	0	400	0	0.8
Hexanethiol-Heptane (Inh-G)	<0.1	Injected in gas phase	0	400	0	0.8
Commercial Inhibitor (Inh-G)	~0.4	Injected in gas phase	0	0	2000	0
Commercial Inhibitor (Inh-G)-HWCR	~0.7	Injected in gas phase	0	0	2000	0

*Inh-G: Inhibitor injected in gas phase, Inh-L: Inhibitor injected in liquid phase

RESULTS

The results of weight loss corrosion rate after the experiments with decanethiol, are presented in Figure 2. As described earlier, in two different experiments, decanethiol was injected into the system through the liquid or the gas phase. According to Figure 2, the results of both experiments show a reduction in the corrosion rate compared to the blank test under similar conditions, which confirms the inhibition provided by decanethiol. In addition, the results of the experiment “Decanethiol (Inh-G)” confirms the transport of the inhibitor from the pre-bubbler to the main glass cell through the gas phase and the effectiveness of this injection method. It can be concluded that the inhibition efficiency of decanethiol is not dependent on the injection method, at least in the absence of hydrocarbon.

The result changes when a hydrocarbon phase (n-heptane) is added to the system. Firstly, by comparing the results of the two blank tests, it is noticed that the corrosion rate decreases with the presence of n-heptane in the main glass cell. N-heptane is not corrosive, and its condensation rate is significantly higher than the one of water. Therefore, in the experiment with n-heptane, the surface of the specimen at the top of the line is mostly wet by n-heptane and not by the corrosive condensed water and this decreases the average corrosion rate. However, since some areas of the surface are indeed covered by water, the risk of localized corrosion remains an issue.

According to Figure 2, the presence of n-heptane in the main glass cell decreases the inhibition efficiency provided by decanethiol. This effect is independent of the injection method. It seems that due to a higher solubility, decanethiol would partition in the hydrocarbon phase both when injected through the gas phase or the liquid phase. When decanethiol is injected through the gas phase, even though it is transported from the pre-bubbler to the main glass cell and is present on the surface of the specimen, it seemingly partitions in the condensed hydrocarbon phase (and obviously not the condensed water) and loses its inhibition efficiency. When decanethiol is injected in the liquid phase in the main glass cell, in addition to the partitioning effect occurring on the surface of the specimen, the same phenomenon occurs in the bottom solution (in the main glass cell) and the inhibitor partitions in the hydrocarbon phase in the bulk solution. This decreases the effective concentration of decanethiol in the solution, and it is reasonable to expect a lower concentration at the specimen surface as well.

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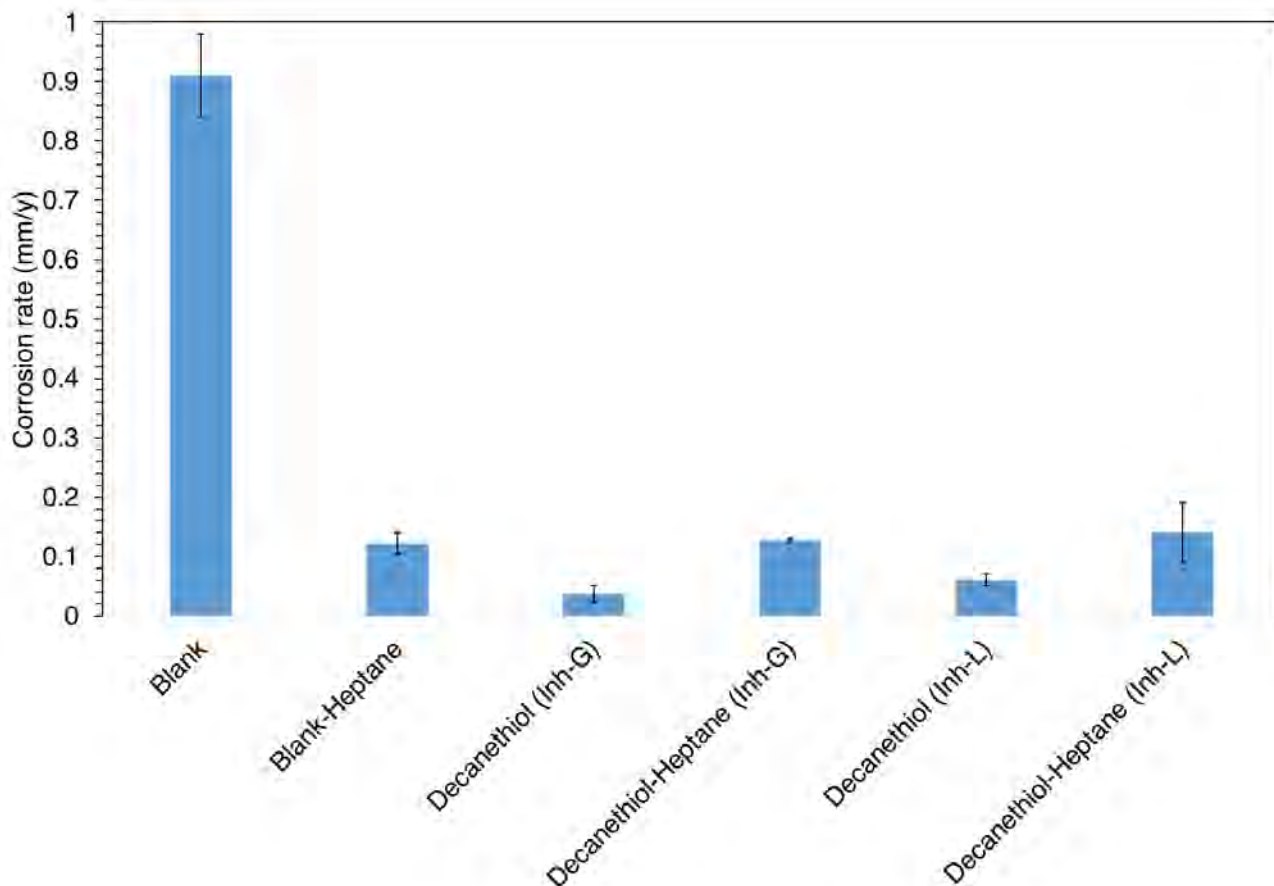


Figure 2: The results of uniform corrosion rate for the experiments with decanethiol (the error bars are minimum and maximum values of corrosion rate).

Figure 3 shows the SEM images of the corroded surface of the specimens after the blank test with and without the hydrocarbon phase (n-heptane) and the three experiments with decanethiol injected through the gas (with and without the hydrocarbon phase in the system) or liquid phase (with and without the hydrocarbon phase in the system).

The surface after the blank tests (Figure 3 (a) and (b)) shows a typical corrosion morphology as a result of selective dissolution of ferrite. When water is the only condensed phase, the addition of decanethiol through the liquid or gas phase significantly protects the surface from corrosion. In this case, the polishing lines are visible on the surface after both experiments “Decanethiol (Inh-L)” and “Decanethiol (Inh-G)” (Figure 3 (c) and (d)). The few pitting-like features on the surface of the specimen in Figure 3 are most probably related to surface preparation before the experiment. The two specimens show minimum differences, with slightly higher corrosion rate for the specimen after the experiment “Decanethiol (Inh-G)” with inhibitor injected through the gas phase (also noted in the corrosion rate results in Figure 2). This small difference comes from the fact that in the experiment “Decanethiol (Inh-L)” with the inhibitor injected in the liquid phase, a higher concentration of inhibitor might be available to protect the surface from corrosion as opposed to the experiment “Decanethiol (Inh-G)”.

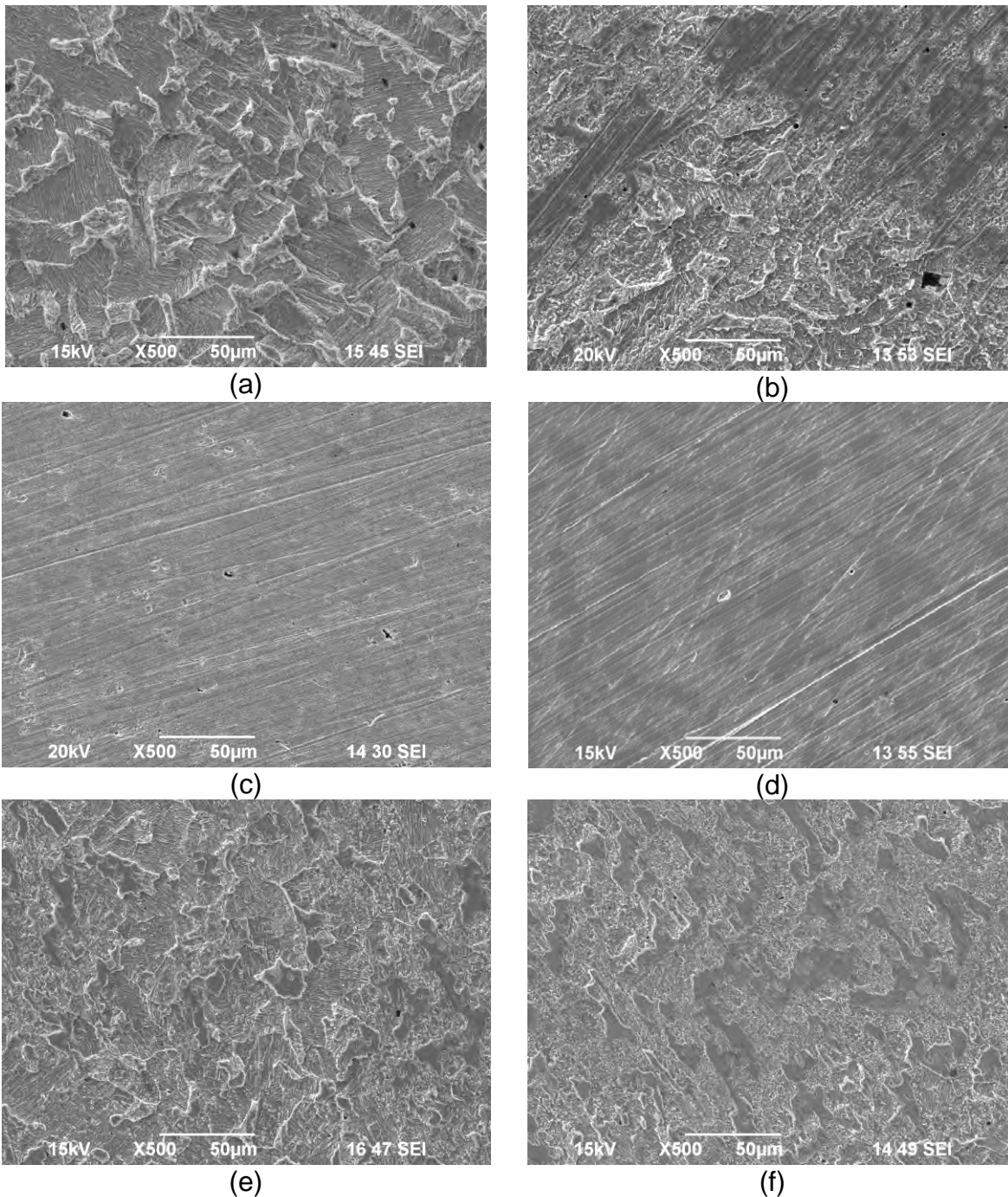


Figure 3: SEM-SE image of the specimen after (a) Blank Test, (b) Blank Test-Heptane, (c) Decanethiol (Inh-L), (d) Decanethiol (Inh-G), (e) Decanethiol-Heptane (Inh-L), and (f) Decanethiol-Heptane (Inh-G) (the images are taken after cleaning the surface with Clarke solution).

When the specimen is tested under the similar conditions and in the presence of n-heptane, a more severely corroded surface is observed. This is observed both with decanethiol injected in the liquid phase (Figure 3 (e)) or injected in the pre-bubbler and transported to the surface of the specimen through the gas phase (Figure 3 (f)). This observation confirms the loss of inhibition for decanethiol in the presence of a condensed hydrocarbon phase that was also reflected in the corrosion rate results.

Optical profilometry was performed on these specimens to find any localized corrosion that might have occurred on the surface. The results are shown in Figure 4.

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These optical profilometry results are in agreement with the results of corrosion rate and SEM. Regarding the two blank tests (Figure 4 (a) and (b)), a more uniform corroded surface is observed in the blank test with only one condensed phase (water) and a more localized corrosion with a deeper corrosion in the areas wet by water is observed for the blank test with co-condensation of water and n-heptane.

The two specimens tested with decanethiol (without the hydrocarbon phase in the system) show minimum differences in the optical profilometry results (Figure 4 (c) and (d)). As observed earlier in the corrosion rate results and SEM images, in the presence of n-heptane, decanethiol loses its inhibition efficiency. The two specimens tested with decanethiol in the presence of n-heptane (Figure 4 (e) and (f)) show corroded surfaces similar to that of the blank test with n-heptane (Figure 4 (b)) with no noticeable difference.

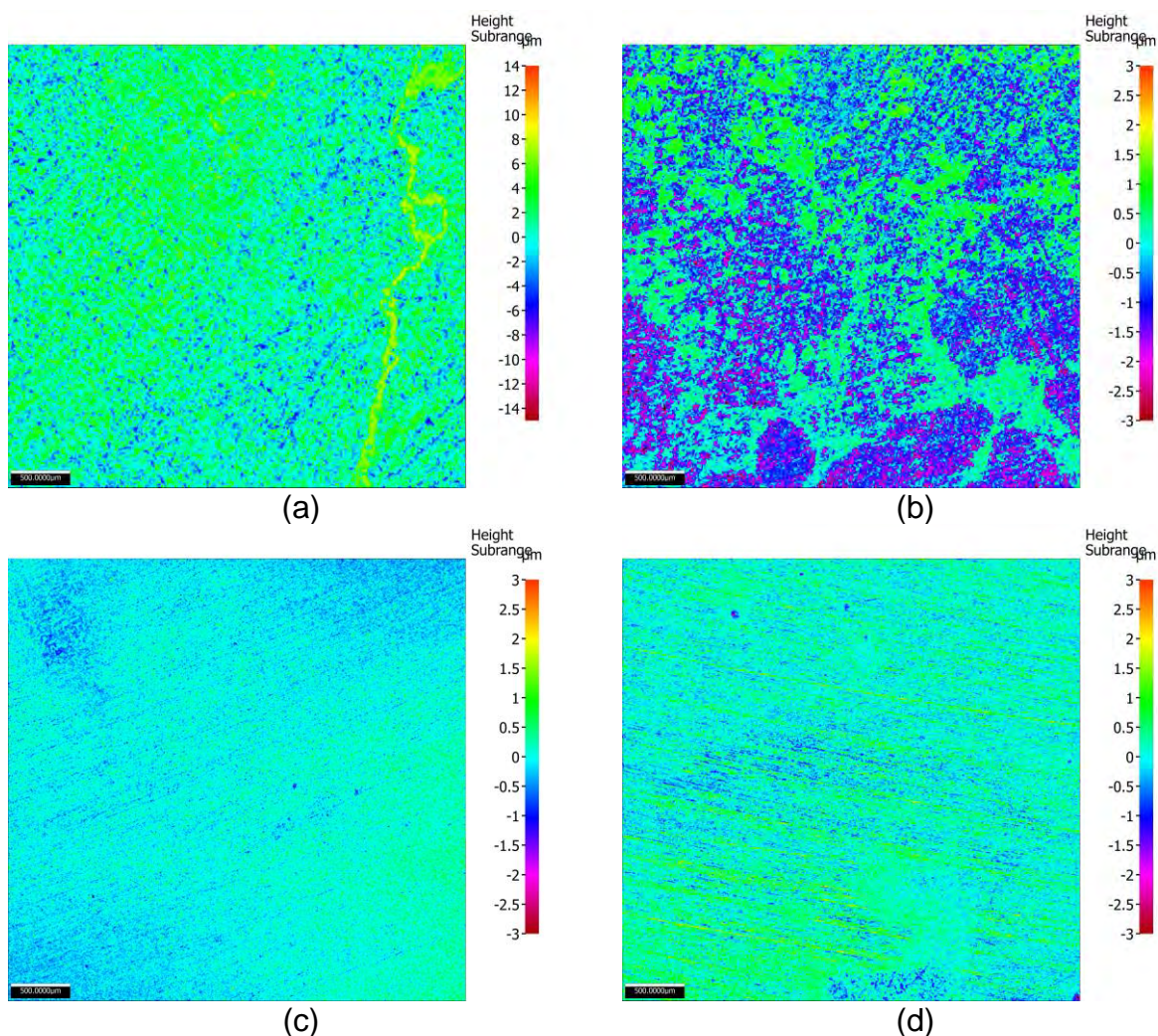


Figure 4: Results of optical profilometry of the sample after (a) Blank Test, (b) Blank Test-Heptane, (c) Decanethiol (Inh-L), (d) Decanethiol (Inh-G)

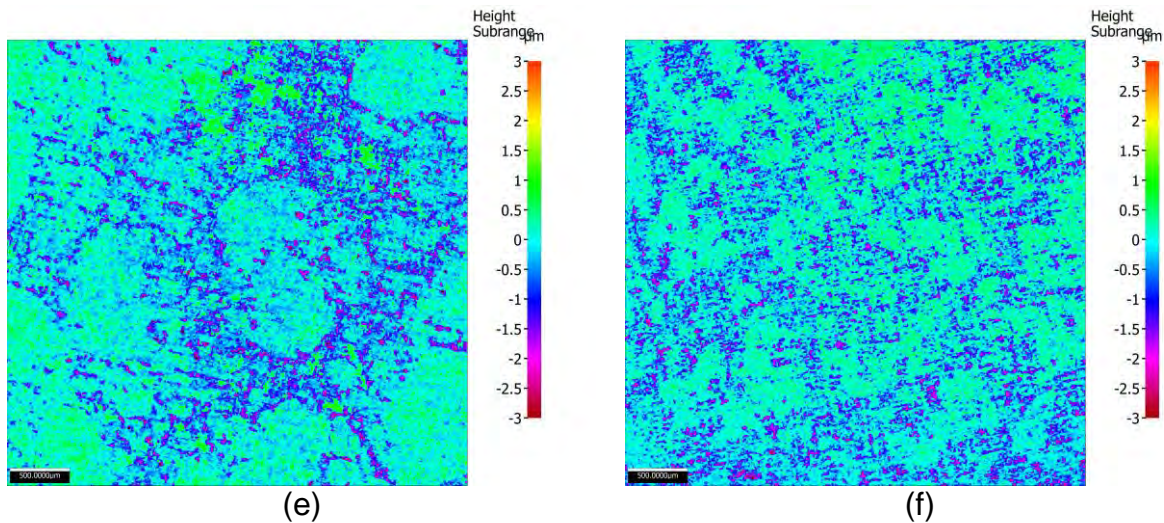


Figure 4 (continued): Results of optical profilometry of the sample after (e) Decanethiol-Heptane (Inh-L), and (f) Decanethiol-Heptane (Inh-G).

In order to further investigate the effectiveness of the injection method in the next experiments, another inhibitor (hexanethiol) was selected. According to a previous study¹⁶, hexanethiol shows a good inhibition even in the presence of n-heptane. The results of experiments with hexanethiol are shown in Figure 5.

According to the results in Figure 5, the addition of hexanethiol through the gas or liquid phase decreases the corrosion rate compared to the blank conditions. Similarly to the results of the experiment with decanethiol, the corrosion rate is higher when the inhibitor is injected through the gas phase, however, the rate is still almost half of that in the blank test and in the “Decanethiol-Heptane (Inh-G)” experiment. This means that hexanethiol is an effective inhibitor in the presence of the hydrocarbon phase.

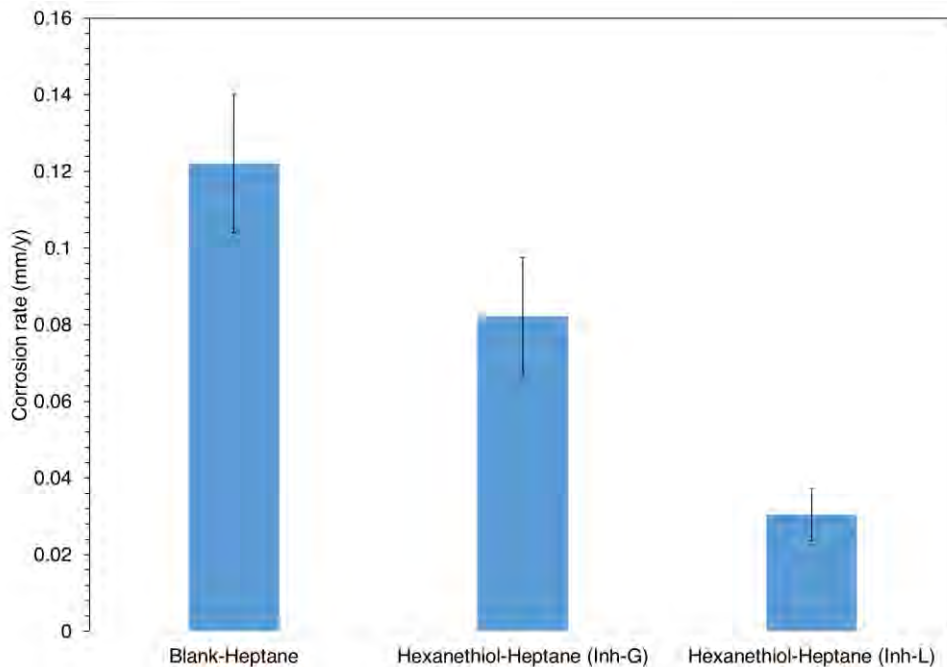


Figure 5: The results of uniform corrosion rate for the experiments with hexanethiol (the error bars are minimum and maximum values of corrosion rate).

The results of SEM analysis and optical profilometry after the experiments with hexanethiol are presented in Figure 6 and Figure 7. As it can be seen in Figure 6, in general hexanethiol protects the surface from corrosion. However, when compared to decanethiol, it obviously provides a less significant protection. This can be understood by comparing the corroded surfaces shown in Figure 6 and those in Figure 3 (c) and (d), where, in the presence of decanethiol, the surface looks almost intact and the polishing lines from the sample preparation step are visible on the surface. In the experiment with hexanethiol, when injected in the liquid phase, the polishing lines are still somehow visible on the surface of the specimen (Figure 6 (a)), however, when the inhibitor is injected through the gas phase, the polishing lines have disappeared and surface looks more corroded. These results agree with the corrosion rate presented in Figure 5. From the SEM images in Figure 6, it can be inferred that the corrosion inhibition provided by hexanethiol is not significantly affected by the injection method.

According to the results of optical profilometry in Figure 7 (with hexanethiol injected in the system through the liquid or gas phase), no severe localized corrosion on the surface of specimens is observed. However, as confirmed by the corrosion rate results in Figure 5, when the transport of the inhibitor through the gas is implemented, a more corroded surface is observed in the optical profilometry data.

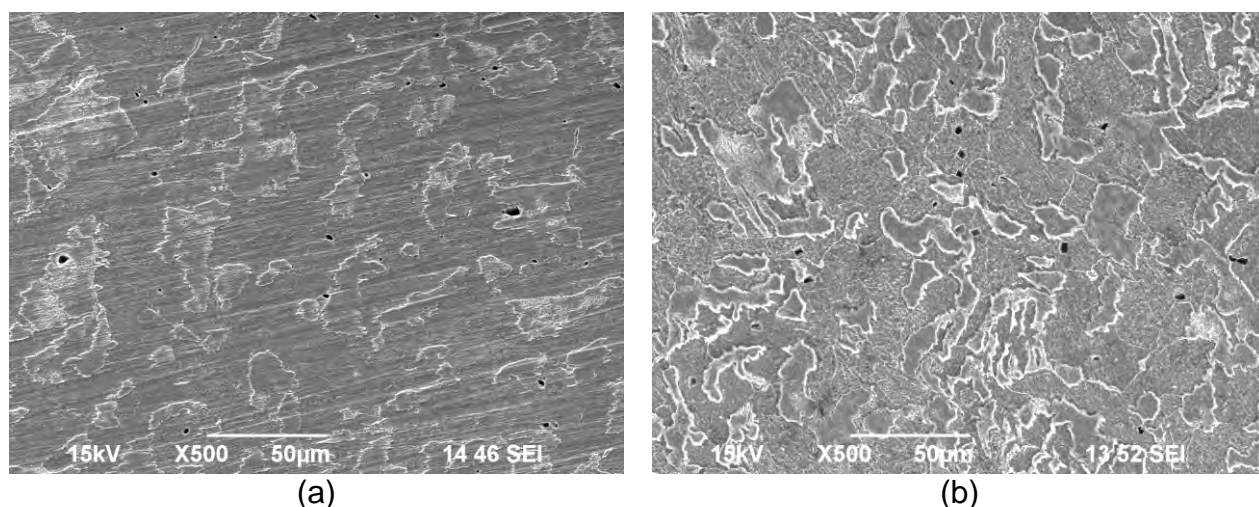


Figure 6: SEM-SE image of the specimen after (a) Hexanethiol-Heptane (Inh-L) and (b) Hexanethiol-Heptane (Inh-G).

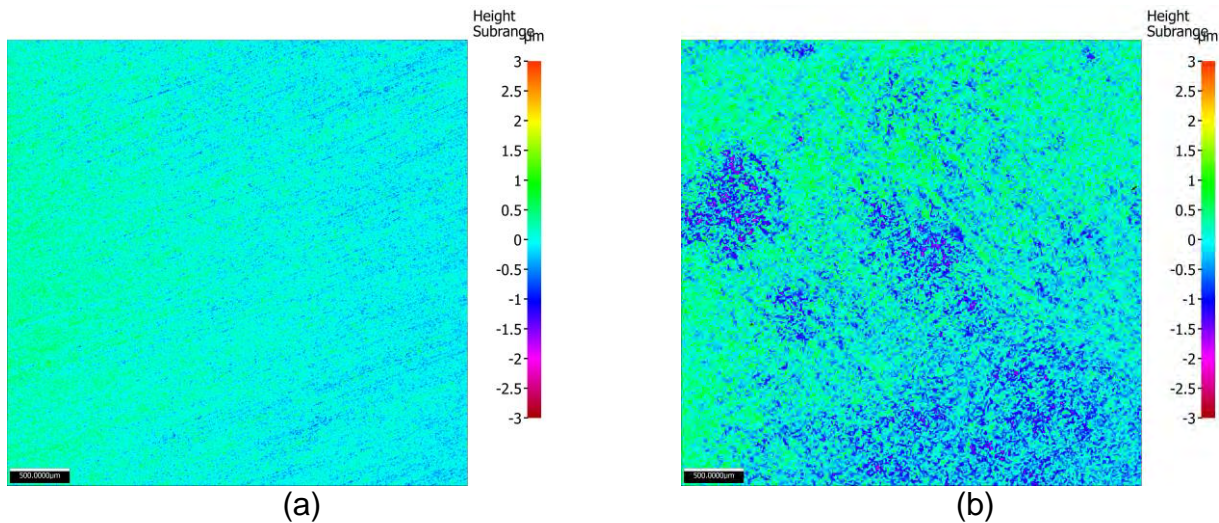


Figure 7: Results of optical profilometry of the sample after (a) Hexanethiol-Heptane and (b) Hexanethiol-Heptane -T.

Since the transport of inhibitor through the gas phase showed some minor effect on the inhibition efficiency of both decanethiol and hexanethiol, two additional experiments with a commercial inhibitor containing amine-based compounds were performed to confirm the validity of the results with VCIs other than model compounds. The choice of an amine-based inhibitor provided the opportunity to measure the concentration of “transported inhibitor” using a method called “methyl orange”²².

These two additional experiments were conducted under two different water condensation rate conditions (0.4 and 0.7 ml/m²/s). The lower water condensation rate condition was similar to that in the previous experiments and the higher water condensation rate was designed for the sole purpose of collecting enough condensed water to enable the measurement of the inhibitor concentration. The recommended dosage for this inhibitor was 2000 ppm, which was injected into the pre-bubbler.

The results of corrosion rate are presented in Figure 8. The corrosion rate of the corresponding blank tests at these condensation rates are also provided in this figure. From these results it can be observed that the selected commercial inhibitor can provide an inhibition efficiency of more than 60% when injected through the gas phase. More importantly, the concentration of transported inhibitor during and at the end of the experiment (after 3 days) could be measured.

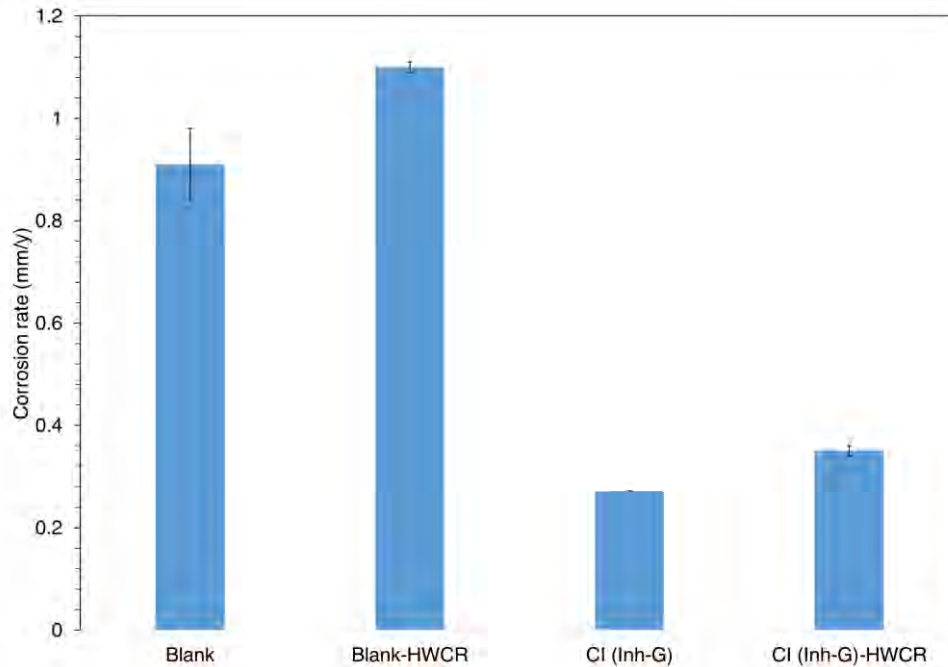


Figure 8: The results of uniform corrosion rate for the experiments with the commercial inhibitor (the error bars are minimum and maximum values of corrosion rate).

In the experiment at a lower water condensation rate ($0.4 \text{ ml/m}^2/\text{s}$), a concentration of 49 ppm of inhibitor was measured in the bulk solution in the main glass cell after three days of experiment. This concentration reflects the amount of inhibitor transported from the pre-bubbler to the main glass cell through the gas phase during the experiment. It should be noted that due to the low volume of collected condensed water during this experiment, the concentration of inhibitor in the condensed water could not be measured. The same measurement was done during and at the end of the experiment with a higher water condensation rate ($0.7 \text{ ml/m}^2/\text{s}$). The methyl orange method measurements showed the concentration of 0.05, 0.8 and 2.4 ppm in the collected condensed water during each day of the 3-day experiment. These values could reflect the amount of inhibitor that is desorbed from the surface of the specimen, nevertheless, the presence of inhibitor in both the condensed water and the bottom solution in the main glass cell confirm the transport of inhibitor from the pre-bubbler and the effectiveness of the implemented injection method. It should be noted that the increase in the concentration of inhibitor in the condensed water with time could be an artifact caused by accumulation of inhibitor in the collection cup. A concentration of 50 ppm of the inhibitor was measured in the bottom solution in the main glass cell at the end of the three-day experiment with a higher water condensation rate (CI (Inh-G)-HWCR).

The results of the experiments with the commercial inhibitor and thiols (with the inhibitor injected through the gas phase) show the effectiveness of this injection method as a TLC mitigation strategy.

CONCLUSIONS

The following conclusions can be drawn from this work:

- An experimental approach was developed to study different methods of inhibitor injection in a CO_2 corrosion system.
- Injection of inhibitor through the gas or liquid phase has minimal influence on its corrosion inhibition efficiency.
- Since the injection method has minimal influence on the inhibition efficiency by an inhibitor, any length of pipe can be protected by an inhibitor injected through the liquid phase as long as the inhibitor is sufficiently volatile.

- Regardless of the injection method, the presence of the hydrocarbon phase (n-heptane) interferes with the corrosion inhibition provided by decanethiol.
- Regardless of the injection method, the presence of the hydrocarbon phase (n-heptane) has no influence on the corrosion inhibition provided by hexanethiol.
- Similar results to those of model compound inhibitors were observed with the selected commercial inhibitor. This commercial inhibitor provides an inhibition efficiency higher than 60% when injected through the gas phase, under the experimental conditions of this study.

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