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Corrosion Resistance Improvement of Mild Steel

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Abstract. Corrosion's devastating consequences have become a big issue all over the world. One of the corrosion damage aspects are related to decontamination solutions which are essential for rapid degradation of toxic agents. The decontamination solutions should be non-corrosive to avoid deterioration of steel container. Decontamination solutions comprising have high chlorinating activity so they have a corrosive effect on mild which are used in fabrication of storage tanks. In this case, using inhibitors is one of the most effective strategies to limit the rate of corrosion. This work describes the effect of different compounds; (zinc chromate, cinnamaldehyde, sodium tripolyphosphate, zinc ortho-phosphate, ammonium molybdate and thiourea) on the rate of corrosion of mild steel within dichloroisocyanuric acid decontamination solution. Meanwhile, these inhibitors should not affect active chlorine content which is responsible for the decontamination process. By using kelthoff and AquaChec® methods for measuring active chlorine, it was found that only zinc chromate passed the tests. For measuring electrochemical behaviour of mild steel in decontamination solutions, different techniques were used. Zinc chromate inhibition on mild steel in a 0.5% dichloroisocyanuric acid solution at different temperatures (298-313 K) were studied utilising electrochemical impedance spectroscopy, weight loss, and potentiodynamic polarisation. The results reveal that zinc chromate is an excellent mild steel corrosion inhibitor in dichloroisocyanuric acid, with an inhibition efficacy of 96% at a concentration of 0.005 M. Finally, surface morphology of mild was also investigated using SEM.

Keywords: Corrosion, Mild Steel, Dichloroisocyanuric Acid Solution and Zinc Chromate.

1. Introduction

Because of its simplicity of manufacturing and inexpensive cost, mild steel has been widely employed in a variety of applications. Mild steel in several applications, such as boiler cleaning, oil well acidification, and pickling, is always into contact with acids. As a result, mild steel is subjected to the corrosion process[1]. Inhibitors are a versatile corrosion prevention strategy for mild steel. To protect mild steel in acidic solutions, many synthetic inhibitors have been utilized; however, because of their negative environmental impact, several of these inhibitors will be phased out soon. Decontamination treatments are critical because they allow harmful chemicals to degrade quickly. They should not only



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be friendly, but they should also not be corrosive. Due to the high chlorinating activity of dichloroisocyanuric acid and its sodium salt, they corrode mild steel, which are employed in storage tanks and pump cores. As a result, using an inhibitor is a good way to slow down corrosion. The use of different inhibitors (zinc chromate, cinnamaldehyde, sodium tripolyphosphate, zinc orthophosphate, ammonium molybdate, and thiourea) to minimize mild steel corrosion rates in various decontamination solutions is discussed in this work. Furthermore, these inhibitors should have no effect on active chlorine, which is important for the decontamination process' efficiency. Zinc chromate has been found to have no effect on active chlorine when measured using AquaChec® and the Kelthoff method. The electrochemical behavior of mild steel in decontamination solutions is measured using a variety of methods. The inhibitory influence of zinc chromate on mild steel in 0.5 percent dichloroisocyanuric acid at room temperature was investigated using potentiodynamic polarisation, weight loss, and electrochemical impedance spectroscopy. The results reveal that zinc chromate is an excellent mild steel corrosion inhibitor in dichloroisocyanuric acid, with an inhibition efficacy of 96 percent at a concentration of 0.005 M. As the concentration of zinc chromate grew, the efficacy of inhibition increased, implying both physisorption and chemisorption mechanisms.

2. Experimental

2.1 Materials

The mild steel specimens were mechanically cut into coupons with dimensions of 5.0 cm x 3.0 cm x 0.1 cm and abraded with fine emery sheets, with a composition (wt.%) of 0.13 percent C, 0.18 percent Si, 0.39 percent Mn, 0.4 percent P, 0.04 percent S, 0.025 percent Cu, and the remainder Fe (400-1200 grade). Before being used in corrosion tests, the coupons were cleaned with distilled water, acetone, and then dried in an oven at 110°C for 30 minutes before being stored in desiccators. Ercros, S.A.'s products provide dichloroisocyanuric acid (DCI). The stock aggressive solution 0.5 percent (by weight) was freshly made by dissolving the acid in distilled water for roughly five hours using a magnetic stirrer. Various inhibitors (ammonium molybdate, sodium tripolyphosphate, zinc orthophosphate, zinc chromate, cinnamaldehyde and thiourea).

2.2 Active Chlorine Number Measurements

Active chlorine is primarily responsible for decontamination solutions' increased corrosive effect on certain metals and alloys, such as crevice and pitting corrosion in high nickel chromium alloys. Active chlorine, on the other hand, is required for the disinfection process mechanism. As a result, it's critical to keep track of active chlorine levels and how time and inhibitor affect them. The iodometric approach was employed in this investigation. In this procedure, 100 mL of acidified decontamination solution is mixed with 10 mL of 0.1 N potassium iodide (KI) (by adding 15 mL of 4 N sulphuric acid). In potassium iodide, chlorine replaces iodine in the decontamination solution. Titrate 0.1 N sodium thiosulfate ($Na_2S_2O_3$) with released iodine (brown color) until straw yellow color appears. When starch is used as an indicator, the color changes to blue, and the sample becomes colorless after further titration. The following equation is used to compute the active chlorine number:

Clorine content (mg. l⁻¹) =
$$\frac{V_1 * N * 35.45 * 1000}{V}$$
 (1)

 V_1 represents the volume of sodium thiosulphate in milliliters, N represents the normality of sodium thiosulphate, and V_2 represents the volume of used decontamination solution in milliliters.

2.3 Weight Loss technique

Because of the ease and consistency of its results, the weight loss approach is a popular approach for determining rates of corrosion. Weight loss studies were carried out in decontamination solutions with varied inhibitor concentrations of 0.0005 M, 0.001 M, 0.0025 M, and 0.005 M at room temperature 298 K, 303 K and 313 K for a total immersion time of 24 hours. Four digital scales were used to weigh the pre-treated steel coupons, which were then immersed in 100 ml of decontamination solution. After the mild steel coupons had been immersed for the required amount of time, they were removed,

washed, dried, and weighed precisely once more. The efficiency of an inhibitor (n) and surface coverage (θ) were estimated utilizing the following equation[2, 3]:

$$\theta = \frac{w_0 - w_1}{w_0}$$
(2)

$$\eta = \frac{w_0 - w_1}{w_0} * 100$$
(3)

Where w_0 signifies weight loss of mild steel without inhibitor, w_i signifies weight loss of mild steel in the presence of inhibitor, θ is the surface coverage and η is the inhibition efficiency.



Figure 1. Corrosion inhibition efficacy of mild steel as a function of concentration (A) ammonium molybdate, (B) sodium tripolyphosphate, (C) zinc ortho-phosphate, (D) zinc chromate, (E) cinnamaldehyde and (F) thiourea inhibitor in 0.5 % DCI solution after 72 h immersion at different temperatures. (G) Variation of mild steel corrosion inhibition efficiency of different inhibitors at 0.01 M concentration in 0.5 % DCI solution for 24 h at 293 K.

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2.4 Potentiodynamic Polarization

The Tafel slopes β_a and β_c , as well as the potential of corrosion, E_{corr} , and the current of corrosion, I_{corr} are all estimated using this potentiodynamic polarisation approach. The Tafel polarisation curves for mild steel as the working electrode were recorded using a potentiostat/galvanostat apparatus type PGP201 in this investigation. As auxiliary and reference electrodes, platinum and standard calomel electrodes were utilized, respectively. All tests were performed at 298±1 K. After immersing the working electrode in the electrolyte solution for 60 minutes, potentiodynamic polarisation curves were acquired. At a scan rate of 1 mV s⁻¹, the electrode potential was automatically altered from -250 to +250 mV vs. corrosion potential during measurements in the decontamination solution containing varying doses of the tested inhibitor. Efficacy of inhibition (η) [4-6] was calculated using:

$$\eta = \frac{i_{corr}^0 - i_{corr}}{i_{corr}^0} * 100 \tag{4}$$

Where i_{corr}^0 : current density of corrosion in without inhibitor, A/cm², i_{corr} : current density of corrosion in presence of an inhibitor, A/cm²

2.5 Electrochemical Impedance Spectroscopy (EIS)

The EIS experiments in this work were completed using a three electrode setup. The reference electrode was a saturated calomel electrode, while the counter electrode was platinum. The EIS tests were conducted utilizing an AC signal at *Ecorr* and a Gamry reference 600 instrument in the frequency range of 100 kHz to 10 mHz with amplitude of 10 mV peak-to-peak. The tests were carried out after mild steel electrodes had been immersed in decontamination solutions for 60 minutes. Efficacy of inhibition (η) [6, 7] was calculated using:

$$\eta = \frac{R_{ct} - R_{ct}^0}{R_{ct}} * 100$$
 (5)

Where R_{ct}^0 : resistance of charge transfer without inhibitor, $\Omega.cm^2 R_{ct}$: resistance of charge transfer in presence of an inhibitor, $\Omega.cm^2$

2.6 Scanning Electron Microscope

A scanning electron microscope (Zeiss EVO-10 microscopy) was used to analyze the surface morphology. In this study, SEM investigated the morphology of the corrosion products formed on the surface of mild steel.

3. Results and discussion

The corrosive media investigated in this work were dichloroisocyanuric acid (DCI) which are used as decontamination solutions. The corrosive effect of these two solutions on mild steel were evaluated. Different inhibitors (ammonium molybdate, sodium tripolyphosphate, zinc ortho-phosphate, zinc chromate, cinnamaldehyde and thiourea) were investigated. During application of inhibitors, one eye should be on the inhibition efficiency and the other on the value of decontamination solution active chlorine which is essential for decontamination process. The weight loss electrochemical techniques were used to evaluate the inhibition efficiency.

3.1 Results of Weight loss technique in DCI solution in the presence of and without different inhibitors Corrosion inhibition efficiency of different inhibitors, namely: ammonium molybdate, sodium tripolyphosphate, zinc ortho-phosphate, zinc chromate, cinnamaldehyde and thiourea, for mild steel are shown in Figures (1A-G) respectively. Measurements of mild steel weight loss immersion in 0.5 percent DCI solution in the presence of and without different inhibitors over 72 hours at various temperatures yielded the results. The efficiency of the inhibition values are: ammonium molybdate 22 %, sodium tripolyphosphate 20 %, zinc ortho-phosphate 27 %, zinc chromate 32 %, cinnamaldehyde 65 % and thiourea 32 %. It is clear from the results that the efficiencies are equal to or less than 35 % except for cinnamaldehyde as shown in figure 1. Consequently, the concentrations should be increased to higher values which may achieve acceptable inhibition efficiency values. The concentrations were raised to 0.01 M as shown in Figure 1(G) for 24 h at 293 K. The efficiency increased to appreciable values (higher than 70 %) for zinc chromate, cinnamaldehyde and thiourea, i.e. 96 %, 74 % and 71 %, respectively. So, these three inhibitors were selected for further investigation. Other inhibitors were rejected either because of low efficiency (sodium tripolyphosphate 58 %, zinc ortho-phosphate 39 %) or limited solubility as ammonium molybdate.

Table 1. Effect of different inhibitors on act	tive chlorine content in 0.5 % DCI solution.
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Corrosive solution	Experimental Time	Volume of sodium	Active Chlorine No.
		Thiosulphate [ml]	
0.5 % DCI	One Hour	7	0.248
0.5 % DCI	24 Hour	3	0.106
0.5 % DCI + 0.01M	24 Hour	3	0.106
Zinc Chromate			
0.5 % DCI + 0.01M	24 Hour	0.2	0.007
Cinnamaldehyde			
0.5 % DCI + 0.01M	24 Hour	0.3	0.011
Thiourea			

3.2 Active chlorine number results

The active chlorine was measured by using both AquaChek® strips and iodometric methods. By using AquaChek® strips shown in Figure (2), the active chlorine numbers for 0.5 % DCI solution in the presence of and without the selected inhibitors (zinc chromate, cinnamaldehyde and thiourea) were evaluated. The comparison of total chlorine color pad of each strip with the total chlorine reference chart is shown in Figure (2) and it is obvious that the free chlorine is not affected by zinc chromate . Iodometric method was used for DCI solutions. It is clear that, concentration 0.01 M zinc chromate has no effect on active chlorine in both acid and salt solutions, respectively. However, it is noteworthy that active chlorine reduced gradually with time for both solutions in the presence of and without zinc chromate as shown in table 1.



Figure 2. AquaChek® package and Total chlorine color pads of 0.5 % DCI solutions (a) in the presence of and without inhibitors: (b) cinnamaldehyde, (c) zinc chromate and (d) thiourea.

At normal temperature, the influence of increasing zinc chromate concentration on the anodic and cathodic polarisation branches of mild steel in 0.5 percent DCI is shown in Figure and table (2). (E_{corr}), Tafel slopes (βc , βa) and (I_{corr}) are determined from extrapolation of the two branches of polarization and stated in Table 3 . From the table, it can be noticed that the corrosion current density decreases with increasing zinc chromate concentration. In addition, the corrosion potential changes within 65 mV. Meanwhile, the cathodic and anodic Tafel slopes were both affected to different extent by the concentration increase of zinc chromate. Zinc chromate operated as a mixed type inhibitor, impacting both anodic and cathodic processes, according to these findings. This is in line with prior studies on zinc chromate as a mixed type inhibitor that has been published.

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Figure 3, Cathodic and anodic polarization curves of mild steel in 0.5 % DCI in absence and presence of different concentrations of zinc chromate at room temperature.

Table 2. Electrochemical parameters for mild steel in 0.5 % DCI solution in absence	e and presence of
different concentrations of zinc chromate at room temperatu	re

Concentration	Icorr[µA.cm ⁻²]	Ecorr[mV]	$\beta_a[mV/decade]$	βc[mV/decade]
0.5 % DCI	39.6	-608.5	492.7	-518.4
0.5 % DCI + 0.001M Zinc Chromate	36.4	-620.8	504.5	-541
0.5 % DCI + 0.0025M Zinc Chromate	9.2	-540.6	365.6	-405.3
0.5 % DCI + 0.005M Zinc Chromate	2.7	-607.1	339.1	-414.6

3.3 Electrochemical impedance spectroscopy (EIS)

The behavior of corrosion of mild steel in 0.5 % DCI solution at room temperature in presence of and without different concentrations of zinc chromate was also investigated by EIS technique. Bode plots of mild steel after one hour and after one-day immersion are represented in Figures (4). It can be noticed that the impedance and phase angle significantly increase by adding zinc chromate. Moreover, they gradually increase by increasing the zinc chromate concentration. In addition, Each Bode plot clearly shows a single inflection in the Bode phase diagram and a single negative value for the Bode magnitude diagram slope, indicating that each of them has only one time constant. Corrosion is primarily regulated by a charge transfer mechanism, as seen by a single inflection in the Bode phase diagram and a single negative value for the Bode magnitude diagram slope. This time constant may be related to electrical double layer at the surface of metal and interface of solution. It is critical to build adequate physical models (equivalent electrical circuits) for impedance spectra that can be used to fit experimental data and extract parameters that define the process of corrosion. To represent the experimental impedance data, Figure 5 illustrates an analogous circuit consisting of a solution resistance, Rs, in series with a parallel combination of charge transfer resistance, R_{ct} , and capacity of doublelayer, C_{dl} .

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Figure 4, Bode plots of mild steel in 0.5% DCI solution in the absence and presence of different concentrations of zinc chromate (A, B) after one-hour immersion at room temperature (C, D) after one-day immersion at room temperature.



Figure 5. Equivalent electrical circuit of electrical double layer formed on mild steel in DCI solutions (in presence of and without inhibitor)

Time	Solution	$R_s [\Omega.cm^2]$	$R_{ct} [\Omega.cm^2]$	Cdl [µF.cm ⁻²]	IE[%]
One	0.5 % DCI	5.4	11.6	59.7	
hour	0.5 % DCI+ 0.001 M inhibitor	7.8	15.3	43.3	24.2
	0.5 % DCI+0.0025 M inhibitor	17.7	105	21.5	88.9
	0.5 % DCI+0.005 M inhibitor	18.5	115	14.3	89.9
One dav	0.5 % DCI	10.4	32.4	127.5	
one duy	0.5 % DCI+ 0.001 M inhibitor	11.04	56.3	60.8	42.5
	0.5 % DCI+0.0025 M inhibitor	13.9	117.2	31.0	72.4
	0.5 % DCI+0.005 M inhibitor	14.6	179.1	27.4	81.9

Table 3. The change in charge transfer resistance and double layer capacitance of mild steel in 0.5 percent DCI solution after one hour and one-day immersion period in the presence of and without different inhibitor concentrations

The change in charge transfer resistance and double layer capacitance of mild steel in 0.5 percent DCI solution after one hour and one-day immersion period in the presence of and without different inhibitor concentrations are shown in table 4. The results reveal that (R_{ct}) values increase dramatically as the concentration of inhibitor increases, which could be due to an increase in the inhibitor's adsorption level on the surface of metal. Furthermore, from an inhibitor-free corrosive solution to one containing 0.005 M zinc chromate, the value of (C_{dl}) drops dramatically. As a result, the cathodic and/or anodic processes are hampered by the adsorbed inhibitor molecules on the mild steel surface. The potentiodynamic polarization results are consistent with these findings. Furthermore, after one day of immersion, (R_{ct}) values are larger than after one hour of immersion. This phenomenon could be caused by two factors. The first is a protective coating generated by the inhibitor, which prevents electrochemical active species from migrating into the mild steel surface. The second is the progressive decrease in active chlorine levels over time. Equation (2) may be used to calculate the efficiency values of inhibitor, which are reported in Table (4)

3.4 Surface morphology

Surface morphologies of mild steel after immersing in 0.5 % DCI solution in presence of and without 0.005 M zinc chromate were investigated using SEM technique as shown in Figures (6A) and (6B), respectively. In the absence of an inhibitor, the alloy surface is severely degraded, showing uniform corrosion with corrosion products covering all metal surface, Figure (6-A). In the presence of 0.005 M inhibitor. It is obvious that corrosion has been slowed. and the mild steel surface is almost free of corrosion products, Figure (6-B).



Figure 6. SEM images of mild steel after immersion in 0.5 % DCI solution for 24 h. (a) in absence and (b) in presence of zinc chromate

4. Conclusion

- The corrosion data from weight loss techniques shows that the corrosion rates of mild steel in DCI are mostly unaffected by sodium tripolyphosphate, zinc orthophosphate, and ammonium molybdate.
- The active chlorine levels of DCI are influenced by thiourea and cinnamonaldehyde, • according to research using the iodometric method of active chlorine testing.
- There is no effect of zinc chromate on DCI's active chlorine levels. •
- For mild steel, zinc chromate may be used as an efficient corrosion inhibitor with an up to 95% inhibition effectiveness.
- There is agreement in the corrosion inhibition of mild steel in solutions containing 0.5% DCI. • as shown by polarisation investigations, weight loss measures, and impedance measurements.
- SEM examination of the metal substrates demonstrates that zinc chromate prevents the corrosion of mild steel in 0.5% DCI.

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