Evaluation of percentage corrosion inhibition efficiency of mild steel with derivatives of oxazolone

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Abstract:

Carbon Steel is one of the most widely used material in the transportation pipelines of Oil and Gas industry. However, they are prone to corrosion in acidic environment. As preventive measure various mitigation techniques are employed to bring a stag to its corrosion, one of which is the use of inhibitors. In the paper, the study of the inhibition properties of two oxazolone derivatives have been reported. The compounds were synthesized at ease and in high yield. The corrosion studies were performed on carbon steel in three different concentrations of HCl (1N, 1.5N and 2.0N) solutions using weight loss coupon (gravimetric) method. 1N HCl solution was found to have the most effective inhibition property for the oxazolone derivatives. % Inhibition efficiency was found to be maximum with 100 ppm of inhibitor concentration. Surface analysis like SEM and SEM-EDX was also carried out to find out the surface morphology of the mild steel in the presence and absence of the inhibitor and concentration of inhibitor. The adsorption of the oxazolones on the mild steel surface was found to obey Langmuir adsorption isotherm. The obtained results revealed oxazolone derivatives as promising effective corrosion inhibitors.

Keywords: Carbon steel, Organic Inhibitors, Corrosion Rate, Gravimetric Method, Oil and Gas industry.

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1. Introduction

In oil and gas industry, the transportation pipelines are in continuous exposure to oil, water and acid and thus have a great tendency to undergo corrosion [1]. Acid solutions are widely used as a pickling agent for steel and also for removal of rust during various stages of oil and gas transportation [2]. Carbon steel, because of its excellent mechanical properties is the most versatile and widely used material in oil and gas industry. However, its low corrosion resistivity has deteriorated its utility [3]. Among the various preventive measures of corrosion, use of organic and inorganic inhibitor is the most popular one worldwide. To avoid metal dissolution during the process inhibitors are widely used. It has been established that organic inhibitors containing N, S and O atoms with conjugated double or triple bond in the system are good corrosion inhibitors [4]. Various researchers established that these molecules positively adsorb on the metal surface forming a protective layer [5-8].

A review on literature reveals the use of amines, amides, aminopyrimidine, imidazolins, thiazolins, aromatic hydrazide etc. derivatives against acid corrosion of carbon steel and was found to be effective [9-11]. The effectiveness of benzimidazole, benzothiazole, pyridine and pyrrole derivatives was also investigated with significant results as corrosion inhibitor [12-18]. Nanomaterials has also played its key role as efficient corrosion inhibitor [19].

In this paper, we present the corrosion inhibitive properties of two oxazolone derivatives namely 4-(2-Methyl-5-oxo-oxazol-4-ylidenemethyl)-benzaldehyde (I) and 2-Methyl-4-naphthalen-2-ylmethylene-4H-oxazol-5-one (II) against acid corrosion on mild carbon steel. The corrosion inhibition properties of these derivatives were studied in three different concentrations of HCl (1.0N, 1.5N and 2.0N) using gravimetric weight loss coupon method. Surface analysis like SEM, SEM-EDX, qualitative analysis like pH determination, UV-visible spectroscopy of the HCl solutions before and after immersion of the inhibited mild steel and adsorption studies were performed on the samples to give promising results.

2. Materials and Methods

2.1. Preparation of Inhibitors

The two oxazolone derivatives were synthesized using previously reported procedure by Sharma et.al, with 90-95% yield [20] as depicted in Scheme 1. Traditional methods may be employed for

its synthesis [21]. Other relevant methods are also known for the synthesis of oxazolones [22,23].
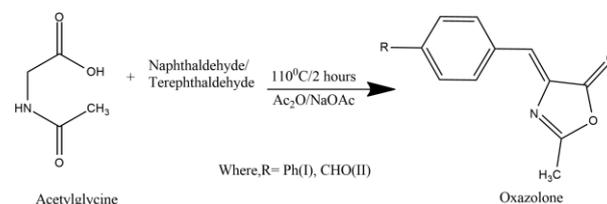
Scheme 1

The synthesized compounds are as follows:
(I):2-Methyl-4-naphthalen-2-ylmethylene-4H-oxazol-5-one;
(II):4-(2-Methyl-5-oxo-oxazol-4-ylidenemethyl)-benzaldehyde

2.2. Sample Preparation

The carbon steel samples (composition: C= 0.28%, Mn =1.4%, P= 0.3%, S=0.3%, V-Nb = Ti $\leq$ 15%) were cut into coupons of dimension 4.5cm x 4.4cm x 0.7 cm with exposed surface area of 19.8 cm2. Before carrying out the experiments the steel coupons were polished with different grades of emery paper, washed with double distilled water several times, subsequently degreased with acetone and finally dried at 60-70 oC. All the solutions were prepared using double distilled water. The chemicals used in the experimental purpose were all analytical grades of Merck.
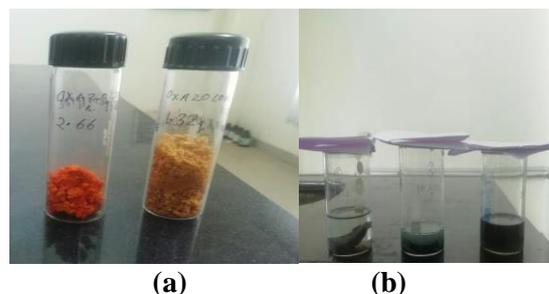
(a)   **(b)**
Fig 1. (a) Synthesized Compounds I And II (b) Weight Loss Coupon Experiment

2.3. Gravimetric Method

In the Gravimetric weight loss measurement, the steel coupons samples were first immersed in 100 mL of three test solutions (1.0N, 1.5N and 2.0N HCl) in uninhibited condition at room temperature and allowed to stand for 48hours. The samples were then coated with inhibitors of concentration 50ppm, 100ppm, 150ppm and 200ppm and immersed in the same test solution for 48 hours. % Inhibition efficiencies (%IE) for different inhibitor

concentration was then calculated from the difference in weight loss before and after addition of inhibitor to the steel specimens using the formula given below [3].

$$IE = \left[\frac{(W - W_i)}{W}\right] X\ 100$$

Where, W= average weight loss in uninhibited condition, and

Wi= average weight loss in inhibited condition

The corrosion rate (CR) is also calculated from the weight loss measurements using the formula given below [3].

$$CR = (K * \Delta W) / (A * t * d)$$

Where, K= constant ($8.76 X 10^4$) that allows to represent CR in mm/yr

A= surface of metal sample (in cm^2)

t= immersion time (hours)

d= density of the metal (g/cm^3)

ΔW = average weight loss

2.4. Scanning Electron Microscopy (SEM)

The steel samples were immersed in the test solution (1N HCl) in presence and absence of inhibitor to inquire the effectiveness of corrosion and inhibition. The steel samples were then dried at room temperature. Micrographs of corroded and inhibited steel surfaces were taken using FESEM instrument (Model Sigma).

2.5. SEM-EDX Spectroscopy

SEM and Energy Dispersed X-ray spectrum imaging for the surface of the studied steel was performed in order to detect elemental composition of the material. The analysis was carried out using FESEM Instrument (Model Sigma). The gold coating was done at "Timed gold" mode with sputter current 20mA and sputter-time 59 seconds.

2.6. UV-Visible Spectroscopy

The test solutions of 1N, 1.5 N and 2N HCl with and without inhibitor, before and after immersion of the steel samples of all four concentrations for 48 hours were subjected to UV-Visible detection using SHIMADZU UV-Visible Spectrophotometer (Model UV1900). The wavelength range was in the UV-visible region, viz. 200-800 nm with medium scan-rate and light source λ equal to 340.8 nm.

2.7. pH Measurements

The pH measurements of the test solutions of concentrations 1N, 1.5N and 2 N HCl before and after immersion of steel samples were conducted using pH meter. pH measurements were also conducted for the test solutions before and after immersion of steel with different inhibitor concentrations.

2.8. Electrical Resistance Measurements

Ohm's law reveals that resistance is inversely related to current. For a material with higher electrical resistance, cross-sectional area decreases and which in turn results in lower corrosion current, indicating lower corrosion rate. To establish the fact, electrical resistances of steel samples in different acid environment both inhibited and uninhibited, electrical resistance measurements were conducted using Multimeter DT830D UNITY model.

3. Results and Discussion

3.1. Weight Loss Measurements

Weight loss for the steel coupons in absence and presence of synthesized inhibitor in the concentration range 50,100, 150 and 200 ppm was calculated in 1.0N HCl, 1.5N HCl and 2.0N HCl. The immersion time period was 48hours for all. All the measurements were performed in triplicate and the average weight loss values were taken for calculation of corrosion rates and inhibition efficiencies. The results obtained are tabulated in table 1 and table 2.

Table 1: Corrosion Properties of Uninhibited Samples

Sample	Solution (in HCl in Normality, N)	Weight loss (in g)	Corrosion rate ($mgcm^{-2}h^{-1}$)
	1	0.86	10.08
Steel Coupon Uninhibited	1.5	1.26	14.51
	2	1.44	16.58

The results from Table 1 and Table 2 showed that in reference to the different concentrations of the HCl solutions, the corrosion rates were found to increase with increase in HCl concentration in the order 1N HCl < 1.5 N HCl < 2N HCl. Thus, the lesser the concentration of HCl in the oil and gas pipelines, lesser will be the rate of corrosion. Further addition of inhibitors decreased the corrosion rate. The % Inhibition efficiency seems to be higher in case of compound I compared to that of compound II implying greater adsorption of compound I on steel surface than compound II. For both the compound % IE increased with increase in inhibitor concentration. The increased adsorption is due to the presence of heteroatoms O and N in the oxazolone molecules, which forms coordinate bonds with the metal surface. Conjugation in the oxazolones is another reason for their increased % inhibition efficiencies. The change in corrosion rate and % inhibition efficiency with change in concentration of HCl for Inhibitor I can be

(a)

(b)

Fig 2. (a) Change of Corrosion Rate and (b) Change of % Inhibition Efficiency of Inhibitor I in 200 ppm I Concentration with Different Concentration of HCl

Table 2: Corrosion Properties of the Samples Coated with Corrosion Inhibitors

Sample	Solution (in HCl in Normality, N)	IC* (in ppm)	Weight loss (in g)	CR* (mg cm^{-2}h^{-1})	% IE*
Steel Coupon with Inhibitor I	1	50	0.29	3.34	67.05
		100	0.12	2.09	86.05
		150	0.06	0.66	93.02
		200	0.02	0.29	97.67
	1.5	50	0.44	5.07	65.07
		100	0.32	3.68	74.60
		150	0.11	1.09	91.26
		200	0.06	0.60	95.23
	2	50	0.64	7.37	55.52
		100	0.44	4.43	69.44
		150	0.10	2.13	93.05
		200	0.08	1.06	94.44
Steel Coupon with Inhibitor II	1	50	0.31	3.57	64.20
		100	0.22	2.21	74.41
		150	0.19	1.98	77.90
		200	0.11	1.32	87.20
	1.5	50	0.64	7.41	48.90
		100	0.45	4.88	64.28
		150	0.29	3.05	76.98
		200	0.21	2.21	83.33
	2	50	0.81	9.33	43.75
		100	0.56	4.99	61.11
		150	0.32	4.30	77.78
		200	0.27	3.11	81.25

(a)

(b)
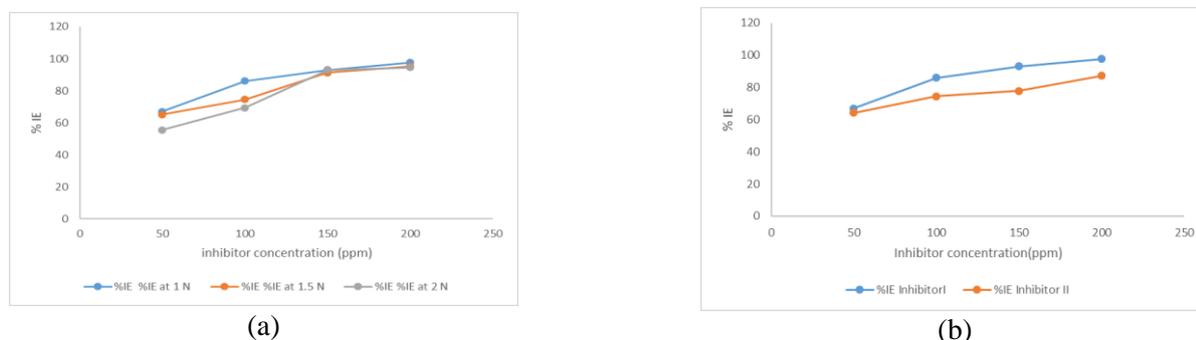
Fig 3. (a) Change of %IE with Inhibitor Concentration for Inhibitor I. (b) Comparison of the Inhibitor Efficiency of Inhibitor I and II in 1N Concentration of HCl

explained more precisely in the figure 2a and 2b shown below. Figure 2 (a) shows that the rate of corrosion increases with increase in HCl concentration in 200ppm inhibitor concentration of Inhibitor I. Similar will be the case with Inhibitor II. The % inhibition however, is decreased with increase in HCl concentration in 200ppm inhibitor concentration of Inhibitor I as depicted in figure 2 (b).

This shows that both the inhibitors I and II work better in lower HCl concentration. Comparison of the inhibition efficiency in 1N HCl test solution for both inhibitors I and II is shown graphically in figure 3. With increase in inhibitor concentration inhibition efficiency was found to increase being highest in case of 200 ppm concentration. In case of 1.5 N HCl and 2N HCl similar pattern was observed.

Further, it was observed that the corrosion rate decreases with increase in Inhibitor concentration. For both inhibitors I and II, similar trend was observed. This increase in the corrosion rate with change in the Inhibitor concentration is graphically represented below in figure 4 for inhibitor I in 1N HCl test solution. For inhibitor I in 200 ppm inhibitor concentration, the corrosion rate was considerably low compared to the high values of corrosion rate in 50 ppm inhibitor concentration. Similar observation was observed for inhibitor II. On comparing Inhibitor I and II, I exhibited minimal corrosion rate, thereby showing its potential as efficient corrosion inhibitor.
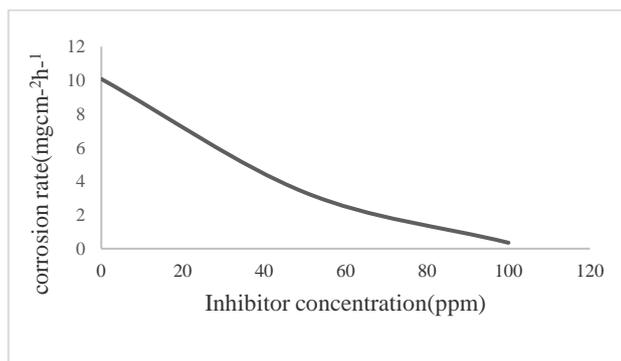
Fig 4. Plot of Inhibitor Concentration vs Corrosion Rate for Inhibitor I

3.2. Electrical Resistance Measurements

The resistances in ohm offered by the steel samples under different environments of test solutions as measured by Multimeter DT 830D UNITY is tabulated in table 3.

Table 3 shows that as the inhibitor concentration increases from $0 \rightarrow 50 \rightarrow 100 \rightarrow 150 \rightarrow 200$, the electrical resistance also increases. Again resistance increases with decreased cross-sectional area of the metal, which in turn is directly proportional to the corrosion rate. Thus, the rate of corrosion decreases with increased electrical resistance. The observed results satisfy the concept and hence it is implied that the compounds I and II show good scope as corrosion inhibitors for oil and gas pipelines. The electrical resistance is the highest with 200 ppm inhibitor concentration and it showed the same pattern with the coated steel sample in all the three concentrations of the test solution. The variation of Electrical resistance with inhibitor concentration after the coated steel

sample are immersed in the test solutions of three different concentrations are depicted vividly in figure 5 below.

Table 3: Electrical Resistance Measurements for Steel Sample Coated with Inhibitor I

Test solution concentration	Inhibitor concentration (ppm)	Resistance in ohm
1 N	0	2
	50	3.5
	100	4.2
	150	5.0
	200	8.3
1.5 N	0	1.6
	50	3.1
	100	6.0
	150	7.7
	200	8.4
2 N	0	1.3
	50	2.0
	100	3.0
	150	3.6
	200	5.0
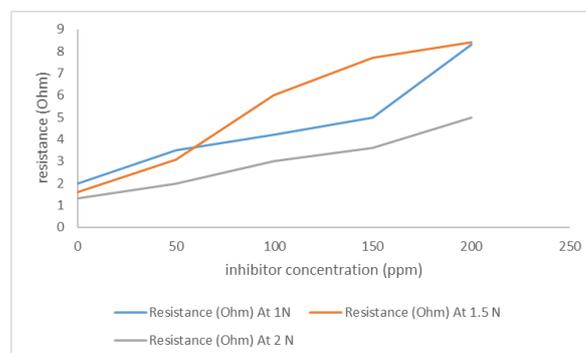
Fig 5. Variation of Electrical Resistance with Inhibitor Concentration

3.3. pH measurements

The pH of the HCl test solutions before and after immersion of the inhibitor coated steel samples were recorded. The results obtained are tabulated in table 4.

Table 4: pH Measurement of Test Solution Before and After Addition of Coated Steel Samples for Inhibitor I.

Test solution concentration	Inhibitor concentration (ppm)	pH before immersion of sample	pH after immersion of sample
1 N	0	0.6	1
	50	0.6	0.7
	100	0.6	2.9
	150	0.6	2.7
	200	0.6	2.3
1.5 N	0	0.2	0.6
	50	0.2	0.6
	100	0.2	2.1
	150	0.2	2.7
	200	0.2	1.1
2 N	0	0.1	0.2
	50	0.1	0.5
	100	0.1	0.8
	150	0.1	2.2
	200	0.1	1

The pH measurement results showed that pH increases considereably with increase in the inhibitor concentration after imersion of the steel sample coated with inhibitor I. Before coating the pH was constant for the test solutions. The variation in pH of the inhibitor coated steel samples was due to the uneven dissolution of the inhibitor compund in the HCl test solutions when the solution was allowed to stand for 48h.

3.4. UV-Visible Spectroscopy

UV-Visible studies of the test solutions before and after immersion of the coated samples were studied and the results obtained was tabulated in table 5. The UV-Visible studies revealed that the increased λ_{max} of the inhibitor coated steel samples were due to the slight dissolution of the coated inhibitor in the test solutions of three different concentration.

3.5. Scanning Electron Microscopy (SEM)

Scanning Electron Microscope photographs suggested the comparatively less corroded surface of steel after inhibitor coating. The inhibition action is due to film formation through adsorption on the steel surface. The morphology of steel in figure 6(a) is for corroded surface without inhibitor having pits and crack on it. Figure 6(b) and 6(c) shows the morphology of inhibited steel with inhibitor compound I and compound II respectively at 200 ppm concentration having less corroded and smooth surface. The thickness of the coating ranged from 50-200Å as observed from the SEM analysis.
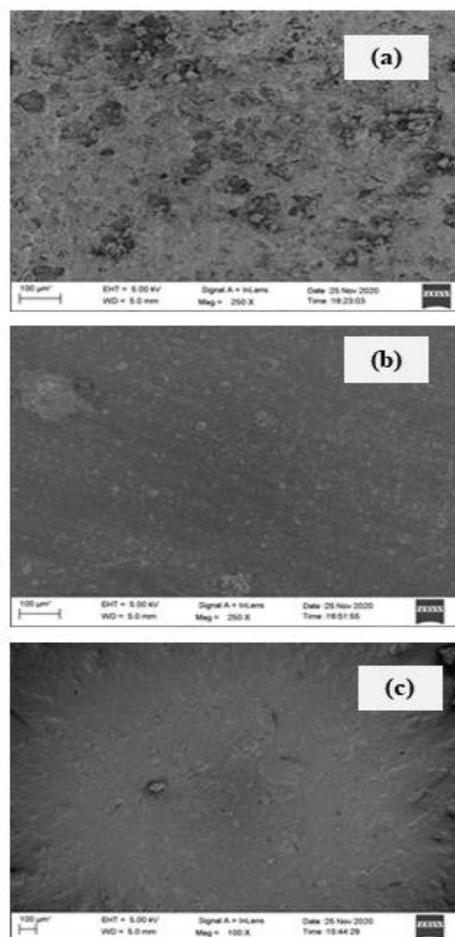
Fig 6. (a) SEM Micrograph of Uninhibited Steel (b) SEM Micrograph of Steel Coated with Inhibitor I. (c) SEM Micrograph of Steel Coated with Inhibitor II

2. Material and Methods

Table 5: UV-Visible Studies for Test Solutions

Test solution concentration	Inhibitor concentration (ppm)	Before immersion of coated sample		After immersion of coated sample	
		λ_{max}	Absorbance	λ_{max}	Absorbance
1 N	0	211	-1.062	334	0.60
	50	211	-1.062	290	2.27
	100	211	-1.062	312	1.14
	150	211	-1.062	314	0.87
	200	211	-1.062	315	1.26
1.5 N	0	211.5	-1.039	329	0.94
	50	211.5	-1.039	292	0.99
	100	211.5	-1.039	314	1.68
	150	211.5	-1.039	314	1.54
	200	211.5	-1.039	322	1.24
2 N	0	212	-0.996	332	1.46
	50	212	-0.996	293	0.99
	100	212	-0.996	315	1.12
	150	212	-0.996	315	1.27
	200	212	-0.996	322	1.24

3.6. SEM-EDX Analysis

The SEM-EDX of the uninhibited sample (figure 7a), steel sample inhibited with inhibitor I (figure 7b) and with inhibitor II (figure 7c) are depicted below. Using elemental mapping in the SEM and EDX spectrum imaging (EDXSI), EDX can detect even trace quantities of elements within a material. The elemental concentration in the spectrum of the uninhibited sample portray the maximum concentration of the elements present in the bare steel samples.
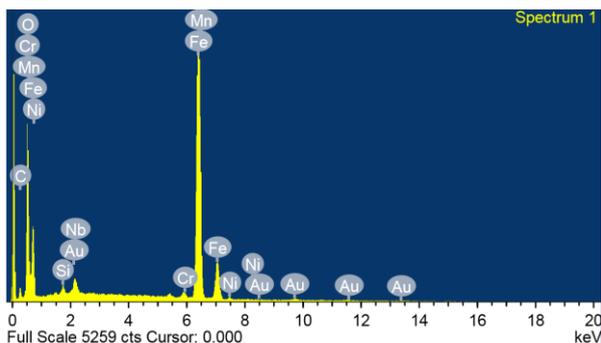
Fig 7. (a) SEM-EDX Spectrum for Uninhibited Steel
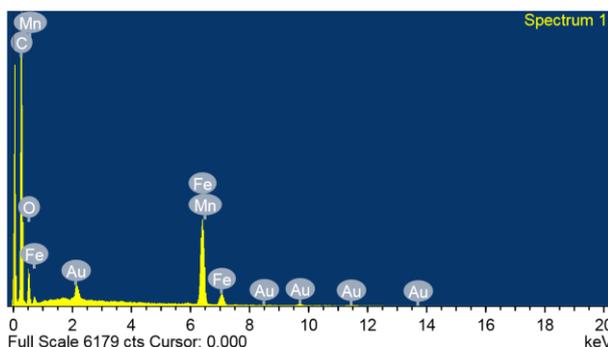
Fig 7. (b) SEM-EDX Spectrum for Steel with Inhibitor I
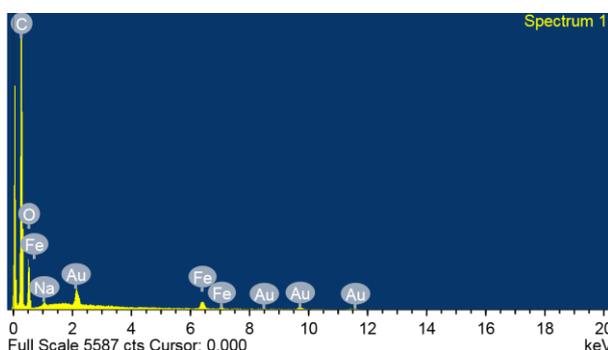
Fig 7. (c) SEM-EDX Spectrum for Steel with Inhibitor II

The SEM-EDX spectra of the steel samples coated with 200 ppm of Inhibitor I and II showed high concentration of C and O, indicating C-O framework

of the coated organic inhibitor. The atomic % of C and O in the spectra for steel coated with inhibitor I together covered ~91% of the total elemental concentration. Same is with the spectra of the steel sample coated with inhibitor II which holds ~98% of the total elemental concentration. Thus the EDX results revealed the concentration of the inhibitor coating which formed a thin film on the surface of the steel sample.

3.6. Langmuir Adsorption Isotherm

The adsorption behavior of Oxazolone derivatives on the steel surface was analyzed by employing Langmuir Adsorption Isotherm to fit the experimental data obtained by weight loss. The isotherm which is a plot of log C/ θ against log C is given by the following equations [24]

$$C/\theta = 1/K_{ads} + C \text{ ----------- (1)}$$

$$\theta = \%IE/100 \text{ --------------- (2)}$$

Where, θ is the surface coverage, C is the concentration of inhibitor and Kads is the adsorption equilibrium constant. The value of Kads as calculated from equation (1) is found to be 0.8714 ppm. Figure 8 showed a plot of C/ θ against C for inhibitor I which is a straight line with slope almost equal to 1 and R2 equal to 0.9998. It confirms the adsorption behavior of oxazolone derivatives on steel surface which obeys Langmuir adsorption isotherm.
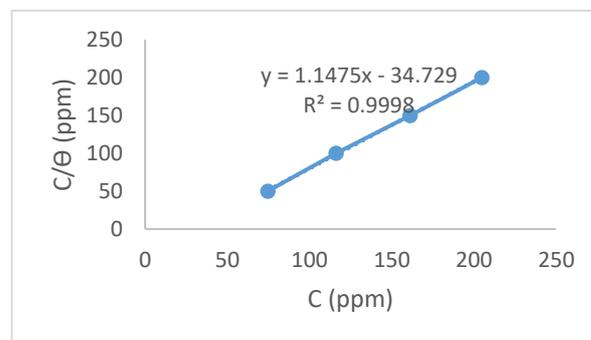
Fig 8. Langmuir adsorption isotherm for Inhibitor I on X60 steel at 1N HCl concentration

4. Conclusions

Organic inhibitors are efficient corrosion mitigators in oil and gas industry. In this paper, we report two oxazolone derivatives, synthesized by previously reported procedures and their inhibiting properties for carbon steel in three varying HCl concentration was studied. 1N HCl concentration showed significant result in terms of % corrosion efficiency for both the inhibitors I and II. Compound I was found to be more effective than compound II with an % IE of 97.67% with 200ppm inhibitor

concentration. The decreased corrosion rate also validates the said statement of inhibitor I. The results of Gravimetric analysis and Electrical resistance corrosion monitoring methods validated the decreased corrosion rate on application of the inhibitors on the steel samples. Surface studies viz SEM, SEM-EDX and Langmuir Adsorption further authenticated the positive aspects of the inhibitor coatings. Further UV-Visible and pH studies of the test solution with and without inhibitor coated steel revealed slight dissolution of the inhibitor in test solutions. Thus, this work shows great scope for oxazolone derivatives and the molecules with similar structural framework to act as effective corrosion inhibitors in oil and gas industry. Oxazolones proving as efficient corrosion inhibitor for mild steel can be blended with highly colored plant extracts for promoting green technology. This in turn will meet the needs of corrosion control without causing any harm to the environment.

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