Indian Journal of Chemical Technology
Vol. 30, January 2023, pp. 117-123
DOI: 10.56042/ijct.v30i1.63539

Experimental investigation of new compound adsorption on carbon steel in 1M HCl

Hojat Jafari[1], Fariba Soltanolkottabi*, [1], Mohamad Mostafa Behrouzfar[2], Elham Ameri[1] & Iman Danaee[3]

[1]Department of Chemical Engineering, Shahreza Branch, Islamic Azad University, P.O. Box 311-86145 Shahreza, Iran

[2]Department of Mechanical Engineering, Faculty of Montazeri, Khorasan Razavi Branch, Technical and Vocational University (TVU), Mashhad, Iran

[3]Abadan Faculty of Petroleum Engineering, Petroleum University of Technology, Abadan, Iran

E-mail: f_kottab@iaush.ac.ir

Received 24 May 2022; accepted 23 November 2022

The effect of new compounds named, 6,6'-(((2,2-dimethylpropane-1,3-diyl)bis(azanediyl))bis(methylene))bis(2-methoxyphenol) (*RSH)* on the corrosion inhibiting of ST- 37 low carbon steel in 1.0 M HCl has been studied. The inhibitor effects on the corrosion behaviour of the samples have been determined at three different concentrations, 0.5, 1.0, 1.5 and 2.0 mg/L. Weight loss, polarization curves, AC impedance measurements and Atomic Force Microscopy (AFM) have been utilized to study the corrosion behaviour of carbon steel in corrosive environment in the presence and absence of new ligand. Results show that the inhibition occurs through adsorption of the inhibitors molecules on the metal surface. The inhibition efficiency was found to increase with increasing inhibitor's concentration. Polarization data indicated that this compounds act as mixed-type inhibitors. The corrosion efficiency of RSH at an optimal concentration of 2mg/L is 86. The adsorption of inhibitors follows the Langmuir isotherm. The values of free energy of adsorption in the presence of the corrosion inhibitor are around -32 KJmol^{-1}, which indicate chemiosorption of the molecules. Powerful Atomic Force microscopy is used for the surface morphology studies.

Keywords: Adsorption isotherm, Corrosion inhibitor, Corrosion rate, Free energy of adsorption, Ligand, Synergistic effect, Synthesis

The rapid development of the industry at the present time especially the steel manufacturing industry also spur the development of basic material manufacturing technology such as steel[1-3]. Given the conditions, sufficient mechanical properties, so that the life span can be increased. St 37 steel equivalent to AISI 1045 is widely employed in a variety of applications, such as the construction of metallic structures[4,5]. Because of its low cost, strong mechanical strength and quality, steel is perceived to be the industry's favourite alloy[6,7]. However, this metal is especially susceptible to acidic conditions, since their resistance to corrosion is extremely low in acidic solutions[8]. It corrodes heavily when exposed to an acidic environment and causes a serious economic loss. Today, there are several methods to assess the intensity and nature of the corrosion process[9-11]. In practice, the following methods are most common: gravimetric, electrical resistance, and linear polarization. The application of inhibitors is one of the most effective methods for corrosion control. Corrosion inhibitors are reagents that minimize or prevent corrosion when they are added at low concentrations to an aggressive environment[6,12-14]. The corrosion inhibition process is associated with a change in the state of the metal surface due to adsorption of the reagent or the formation of poorly soluble compounds with metal cations. Inhibitors can act in two ways: reduce the active surface area and/or change the activation energy of the corrosion process[15,16]. Corrosion inhibition occurs by the adsorption of inhibitor molecules or ions on the metal surface to form a protective layer that reduces or prevents metal corrosion. The effectiveness of inhibitors depends on the rate of their adsorption and the ability to form coatings on the metal surface[17, 18]. Furthermore, the adsorption of inhibitors depends on the chemical structure of the inhibitor molecule, electrolyte composition, temperature, nature of the metal surface, and potential at the metal-solution interface. The adsorption of the corrosion inhibitors can be described by the isotherm. The type of adsorption isotherm for each inhibitor is determined by experimentation[19-23]. In recent years, we have prepared some new ligands containing heteroatoms with various applications such as anti-corrosion[13, 24, 25], catalyst[26,27]; antibacterial[28,29], anticancer[30,31], antifungal[32], DNA binding[33] and chemsensor[34]. Chemical compounds containing aromatic rings and heteroatoms (typically nitrogen, sulfur, and oxygen atoms) can be adsorbed on the metal surface. Therefore, they act as corrosion inhibitors and have corrosion inhibitory properties[35, 25]. The first stage of inhibition by these reagents is adsorption at the interface of the metal-solution and the replacement of the inhibitor molecules with water or acid molecules. Then, a chemical bond is formed between the adsorption center of the molecule and the unfilled orbital d in iron[36]. Nitrogen-containing surfactants such as imidazoline and its derivatives have been used successfully to prevent the corrosion process. These organic compounds, by adsorbing on the surface of the metal and forming a barrier layer, prevent the invading ions from reaching the metal surface[37, 35]. Jafari *et al.*[20] reported the Corrosion inhibition effect of 2-mercaptobenzothiazole (MBT) and 2-aminobenzothiazole (ABT) compounds on ST-37 carbon steel in 1 M hydrochloric acid solution by use of electrochemical impedance spectroscopy (EIS). It was observed that both of these compounds have corrosion inhibition effect on carbon steel. The result of this study showed that by increasing the immersion time, corrosion resistance of samples is increased. Sayin *et al.*[21] studied three imidazoline derivatives for corrosion prevention of the carbon steel samples in 1M HCl. three compounds are mixed type inhibitors and inhibition efficiency increases with increasing concentration of compounds. Further, the 4-AT, displayed the best inhibiting characteristics for this system, with a maximum *IE* of approximately 93% having added only 2×10^{-3} M. Jafari *et al.*[22] investigated the inhibition and adsorption of Shiff-base on carbon steel samples in a 1 M HCl solution. They noticed that the inhibition occurred through adsorption of the inhibitor molecules on the metal surface. The inhibition efficiency was found to increase with increasing inhibitor concentration and decreased with increasing temperature. Thermodynamic parameters for adsorption and activation processes were determined. Mohsenifard *et al.*[23] evaluated the corrosion inhibition efficiency of N,N′-Bis(phloroacetophenone)-1,2-propanediamine in HCl solution at various concentrations of carbon steel samples. They observed the corrosion protection was highly dependent on concentrations of the inhibitor in acid. Also, the ligand was adsorbed directly on the carbon steel samples.

In this work, it is aimed to examine the corrosion rate and inhibition performance with and without the use of RSH in a 1 M HCl solution. For this purpose, the weight loss, polarization curves and AC impedance measurements for carbon steel samples is used to determine the optimal concentrations of the inhibitor. The adsorption isotherm of the corrosion inhibitor on the carbon steel samples is investigated to determine the equilibrium constant and standard free energy. Finally, the surface morphology of the sample in 1M HCl with inhibitor and without inhibitor is studied using Atomic Force Microscopy (AFM) method.

Experimental Section

The first step in all corrosion tests is preparing of the test samples. Square carbon steel samples with an area of 1 cm^2 were cooled in cold epoxy resin then polished with grade emery paper number 220 to 2000 and washing them with distilled water and acetone. After that they were dried with air and kept as a working electrode in a desiccator until use. The samples were prepared from ST-37 carbon steel which had the chemical composition (wt. %): 0.11C, 0.56 Mn, 0.03Si, 0.007P, 0.005S, 0.07Cr, 0.03Ni and balance Fe.

The hydrochloric acid test solution was obtained by diluting Merck's product (37% HCl) with distilled water. The concentration of inhibitors employed was 0.5, 1, 1.5, 2mg/L. Figure 1 shows the Schiff base ligand of this study.

Weight loss (mass loss) of mild steel in the 1M HCl solution without and with RSH at 25°C with different immersion period (0 to 14 hours). After the specified time, the steel samples are submerged from

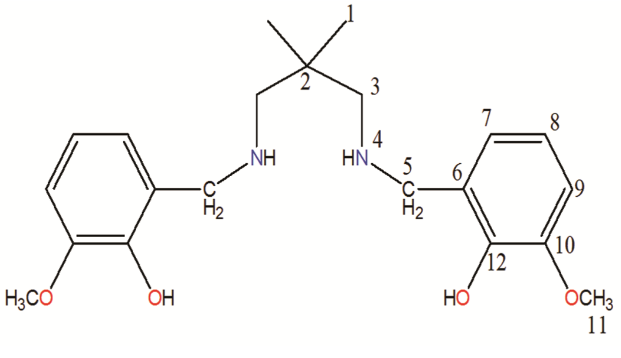

Fig. 1 — Chemical structure of inhibitor

the 1M HCl solution and weight loss of steel sample was recorded. The protection efficiency of the corrosion inhibitor is calculated from the below equation[20],

Corrosion inhibition efficiency (%)

$$= (W_1 - W_2)W_1 \times 100 \qquad \ldots (1)$$

Where, W_1= Unprotected steel weight loss, and W_2=Protected steel weight loss of the system.

Autolab device was used to perform polarization tests. The tests were performed on a standard cell containing platinum wire as an auxiliary electrode and calomel as a reference electrode. The experiments were performed at room temperature. At the beginning of each experiment, an interval of about 30 min was applied to stabilize the potential of the components in solution. From the potential range of about -800 to -200 mV compared to the open circuit potential and scan rate of 1 mV / s.

Electrochemical test was performed for steel samples after 30 min of immersion in the solutions. For this purpose, a three-electrode electrochemical cell including counter electrode (graphite), reference electrode electrode (Calumel electrode) and working electrode (steel sample) is connected to a computer-controlled AutoLab potentiostat / galvanostat system (PGSTAT 302) and tested in open circuit potential in the frequency range of 100kHz to 10mHz with a peak amplitude of 10 mV A.C. was done.

Atomic forced microscopy was used to study the morphology of the samples surface. In this method, steel samples were immersed for 24 h in 1M HCl solution without inhibitor and also containing 2 mg of inhibitor at room temperature. The samples were then removed from the solution and we washed them with distilled water and acetone, they were photographed by atomic force microscopy (AFM) NanoSurf easyscan2.

Synthesis

Synthesis of 6,6'-(((2,2-dimethylpropane-1,3-diyl)bis(azanediyl))bis(methylene))bis(2-methoxy phenol) (RSH).

The inhibitor (RSH) was prepared according to the literature methods[24]. To methanolic solution of 2,2'-((1Z,1'Z)-(((propane-1,3-diylbis(oxy))bis(2,1-phenylene))bis(methanylylidene)) bis (azanylylidene)) diethanol (0.741g, 2mmol), excess $NaBH_4$(0.76 g, 20 mmol) was added carefully in solid incremental amounts with stirring. The mixture was stirred for 20 h at room temperature, filtered off and the solution was concentrated to drynessunder reduced pressure. Yield: 78%. For $C_{21}H_{30}N_2O_4$: Anal. Calcd. (%), C: 67.35; H, 8.07; N, 7.48. Found, (%): C, 67.55; H, 8.03; N, 7.67. IR (ATR, cm^{-1}): 3320 ($\upsilon_{\text{N-H}}$), 1610, 1579, 1480 ($\upsilon_{C=C}$). ^{1}H NMR ($CDCl_3$, ppm): δ=1.00 (s, 6H, H-1), 2.49 (s, 4H, H-3), 3.73 (s,6H, H-11), 3.90 (s, 4H, H-5), 6.58 (d, 2H, H-7), 6.69 (t, 2H, H-8), 6.77 (d, 2H, H-9). ^{13}C NMR ($CDCl_3$, ppm): δ 24.4 (c-1), 34.7 (c-2), 53.2 (c-5), 55.9 (c-11), 57.7 (c-3), 111.0 (c-8), 118.8 (c-9), 120.6 (c-6), 123.1 (c-7), 147.2 (c-12), 148.1 (c-10) (Figure 1).

Results and Discussion

Weight loss method

An important tool for preventing corrosion is the use of compounds with high efficiency, low cost, and low toxicity[16]. Figure 2 presents the results of corrosion rate and inhibition efficiency of oleic imidazoline at 25°C and various concentrations. As shown in the figure, the corrosion rate was increased significantly in the absence of the corrosion inhibitor over time. After 14 h immersion of the carbon steel samples in the HCl solution, the corrosion rate was higher than 2.5 mm/y in the absence of the inhibitor. The addition of the inhibitor reduced the corrosion rate. It is related to the adsorption of the reagent on the surface of the carbon steel samples. As can be seen, with an increase in the inhibitor concentration, the corrosion rate was decreased, and the inhibition efficiency was increased, reaching a constant value. This means that the values of the surface coverage and the corrosion efficiency were enhanced markedly

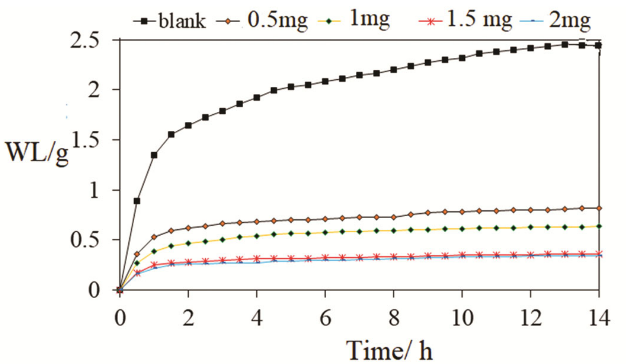

Fig. 2 — Corrosion rate and inhibition efficiency of 2-mercaptobenzimidazole at 25°C.

by increasing the concentration of inhibitor. The optimal concentration of RSH was obtained 2mg/L, as the corrosion rate and inhibition performance were not noticeably changed at higher concentrations. As shown in Fig. 2, the corrosion rate and inhibition efficiency after 14 h at 2mg/L was 87%.

Polarization diagram

Figure 3 shows the polarization diagram of steel in 1M HCl without the presence of inhibitor and different concentrations of inhibitor. Electrochemical parameters including corrosion potential (E_{corr}), corrosion current intensity (I_{corr}) and anodic and cathodic Toefl slopes (β_a, β_c) are measured and are shown in Table 2. The degree of surface coverage (θ) and inhibitory efficiency (*IE* %) are calculated by the following equations[21, 23].

$$\theta = \frac{I° - \mathrm{I}}{\mathrm{I}°} \quad \ldots (2)$$

$$IE\% = I° - \mathrm{II}° \times 100 \quad \ldots (3)$$

$\dot{I}_{corr}$ and I_{corr} are the density of corrosion currents in solution without inhibitor and in the presence of inhibitor, respectively. As shown in Table 2, the corrosion current density decreases with increasing inhibitor concentration. Also, with increasing Schiff base concentration, both tofel anode and cathode slopes do not have a specific trend. This confirms that the inhibitor prevents corrosion by covering the active points on the metal surface[22]. The amount of corrosion potential in the presence of inhibitor is reduced compared to the solution without Schiff base and the corrosion potential decreases with increasing concentration of the inhibitor. Figure 3 shows the results of the polarization test which confirms this. Considering that at low concentration the corrosion potential is reduced compared to the solution without inhibitor. This inhibitor did not have an impressive effect on the anodic and cathodic tofel slopes. It can be concluded that the inhibitory function of Schiff base ligand reduced the exchange rate of anodic and cathodic reactions which has a greater effect on the cathode branch. However, as the inhibitor concentration increases, the corrosion potential moves to more negative values (Fig. 3), which indicates that as the inhibitor concentration increases, its effect on the cathode branch increases. According to Table 1, with increasing the ligand concentration, inhibition efficiency increased. The inhibitor was mixed type due to its effect on the anodic reaction of metal dissolution and the cathodic reaction of hydrogen and oxygen reduction. However, the effect on the cathodic reaction is more pronounced. In other words, the increase in anodic dissolution energy is less than the increase in hydrogen reduction energy. In addition, the corrosion potential changes slightly with increasing concentration, indicating a mixed mechanism[14-17].

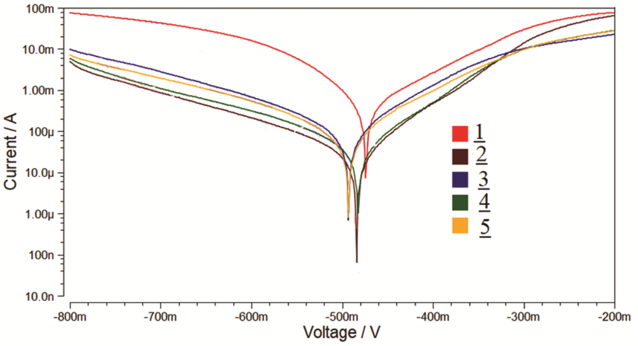

Fig. 3 — Tafel plots without and with corrosion inhibitor: blank (1), 0.5 (2), 1(3), 1.5(4), 2(5) mg/L

EIS test

Figure 4 shows the Nyquist diagram in HCl solution without inhibitor and at different concentrations of inhibitor. As shown in the Fig. 4, the radius of the semicircle increases with increasing concentration. This indicates that with increasing inhibitor concentration, the resistance to electric charge (R_{ct}) increases and, consequently, the degree of corrosion of the steel in solution decreases. The data for the impedance test are shown in Table 2. As the inhibitor concentration increases, the amount of dual layer capacitor (C_{dl}) decreases. This indicates that the inhibitory molecules replace the water molecules on the metal surface. Increasing the load transfer resistance and decreasing the capacitive hardness indicate that the charge transfer is a process that controls the corrosion of steel[36].

Adsorption isotherm of the corrosion inhibitor

The main information on the interaction of the inhibitor with the surface of carbon steel can be obtained from the adsorption isotherm[38]. To obtain isotherms, it is necessary to find linear relationships

Table 1 — Chemical composition of carbon steel electrode used in experiments

Element	C	Si	P	S	Cu	Al	N	V	Ti	Mo	Mn	Cr	Fe
Wt. %	0.18	0.232	0.011	0.002	0.014	0.015	0.076	0.001	0.008	0.001	1.035	0.0009	Rest

Table 2 — Tafel curves results

Concentration, mg/L	$-E_{corr}$ mV	I_{corr} µA cm^{-2}	$\beta_a(\pm 1)$, $mVdec^{-1}$	$-\beta_c(\pm 1)$, mV dec^{-1}	θ $(\pm 10^{-2})$	*IE* %
blank	474	264	79	117	----	-------
0.5	490	70	83	123	0.73	73
1	492	58	66	124	0.78	78
1.5	485	42	87	117	0.84	84
2	480	36	78	120	0.86	86

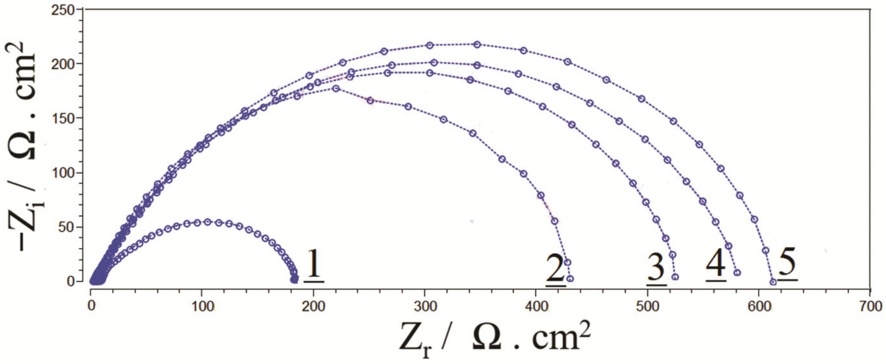

Fig. 4 — Nyquist plots in the absence and presence of the corrosion inhibitor: blank (1), 0.5 (2), 1(3), 1.5(4), 2(5) mg/L

between the values of the surface coverage (θ) and the inhibitor concentration (C_{inh}). The Langmuir isotherm, in contrast to other isotherms, most fully described the adsorption behaviour of the molecules of the studied inhibitors. According to this isotherm, θ is related to C_{inh} by the following equation[38].

$$C_{inh}\theta = C_{inh} + 1K_{ads} \qquad \ldots (4)$$

Where C_{inh} is the inhibitor concentration, K_{ads} is the equilibrium constant of the adsorption process of the inhibitor.

According to Equation 4, K_{ads} can be determined from the intersection point of the straight line on the graph of C_{inh}/θ versus C_{inh}. Also, the standard free energy of adsorption (ΔG°_{ads}) can be represented by the following equation[38].

$$\Delta G^{\circ}_{ads} = -RT\ln(55.5K_{ads}) \qquad \ldots (5)$$

Where ΔG°_{ads} is the standard free energy of adsorption ($J.mol^{-1}$); R is the universal gas constant (8.314 $J.mol^{-1}.K^{-1}$); T is the temperature (K).

As shown in Fig. 5, the plot of C_{inh}/θ versus C_{inh} is a straight line, which depicts that the adsorption of all corrosion inhibitors on the surface of the carbon steel samples obeys the Langmuir isotherm. In this case RSH was tested at 25°C. The coefficient of determination (R^2) was practically equal to unity for all reagents. The high values of the coefficients of determination show that the surface of carbon steel samples was protected by the adsorption of inhibitor molecules, which fully corresponded to the Langmuir isotherm. Moreover, the equilibrium constant of the adsorption process (K_{ads}) is determined according to Equation 4. The results are presented in Table 3. High K_{ads} values indicate that the inhibitor molecules have strong adsorption capacity on the surface of the carbon steel samples. The mixture of inhibitor had the highest K_{ads} value. In addition, the standard free energy of adsorption (ΔG°_{ads}) was calculated using Equation 5 for the compound. The results are also shown in Table 3. Negative values of ΔG°_{ads} are consistent with the spontaneity of the adsorption process and the stability of the adsorbed layer on the

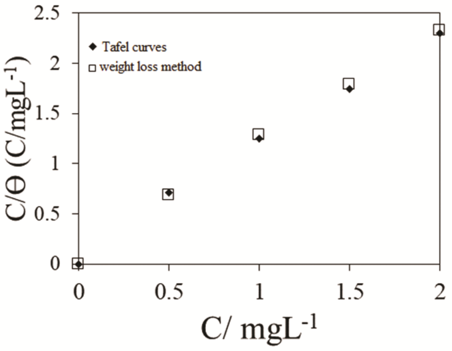

Fig. 5 — Langmuir isotherm adsorption model 4-HCD on the surface of steel in HClsolution: (×) weight loss, () EIS.

Table 3 — AC Impedance studies

Concentration, Mg/L	R_s ($\pm 10^{-1}$), Ω cm^2	R_{ct} (± 1), Ω cm^2	Q_{dl}($\pm 10^{-2}$), mF cm^2	C_{dl}($\pm 10^{-2}$), µF cm^{-2}	n ($\pm 10^{-2}$)
blank	1.4	173	0.216	0.185	0.85
0.5	1.3	423	0.206	0.115	0.81
1	1.4	522	0.185	0.107	0.80
1.5	1.5	584	0.168	0.096	0.81
2	1.6	611	0.082	0.047	0.79

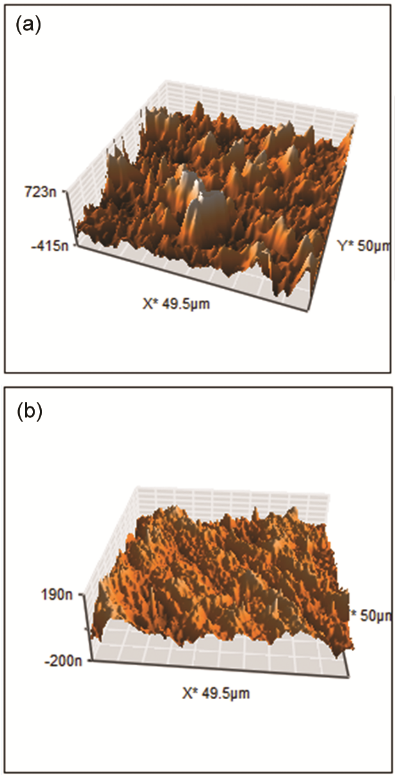

Fig. 6 — (a) AFM images without corrosion inhibitor, (b) AFM images with corrosion inhibitor

surface of carbon steel samples. The values of $\Delta G°_{ads}$ for the corrosion inhibitor was around -32 KJ.mol^{-1}, which indicates that the adsorption mechanism on the surface of carbon steel in 1 M HCl solution corresponds to the chemiosorption of molecules.

Surface morphology study

The morphology of the surface of steel samples in half molar acid solution was examined by Atomic Force Microscopy (AFM) in the presence of 2 mg RSH and without the inhibitor at 25°C and after 24 h of immersion. The results are shown in Fig. 6. In the absence of inhibitor due to rapid corrosion by rough acid solution is observed. Corrosion in this case is relatively uniform and there is no sign of local corrosion. In the presence of the inhibitor, the steel surface roughness is reduced, which indicates the formation of a film on the metal surface and its inhibitory effect.

Conclusion

In this study, the effects of corrosion inhibition and the ability of adsorption of new compound (RSH) on carbon steel surface were investigated and the following results were obtained: This organic compound has inhibition properties in 1M HCl medium.AFM analysis supports the creation of a preventive layer of inhibitor on steel surface layer which facilitates the lowering of corrosion rate. This inhibitor affects both anodic and cathodic branches and reduces the corrosion rate by reducing the exchange current of reactions by blocking existing sites.

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