

Synergistic Effect Between Oleic Imidazoline and 2-Mercaptobenzimidazole for Increasing the Corrosion Inhibition Performance in Carbon Steel Samples

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ABSTRACT: In this work, the corrosion rate and inhibition efficiency of oleic imidazoline and 2-mercaptobenzimidazole in 1 M HCl were studied on carbon steel samples. The corrosion efficiency of oleic imidazoline and 2-mercaptobenzimidazole at a concentration of 75 ppm was 85.8 and 82.9%, respectively. In addition, a mixture of these reagents at a ratio of 1:1 showed enhanced corrosion inhibition. The optimal concentration of the proposed mixture of reagents was 75 ppm. In this case, the inhibition efficiency was 97.8% (by weight loss method). The synergistic inhibition effect between the components of the proposed mixture was obtained 15.9%. Meanwhile, by increasing the temperature from 298.15 to 373.15 K, the corrosion inhibition of the reagents was not considerably decreased, which was associated with their complete adsorption on the surface of the carbon steel samples. The adsorption of inhibitors followed the Langmuir isotherm. The values of free energy of adsorption in the presence of the corrosion inhibitors ranged from -31.6 to -32.62 kJ/mol, which indicated both physisorption and chemisorption features. Moreover, the results of electrochemical impedance spectroscopy and potentiodynamic polarization tests confirmed that the mixture of reagents at the ratio of 1:1 has a significantly higher inhibitory ability compared to each of the reagents alone. The used reagents behaved as a mixed-type inhibitor and affected both cathodic and anodic reactions.

KEYWORDS: Corrosion inhibitor; Adsorption isotherm; Synergistic effect; Free energy of adsorption.

INTRODUCTION

Corrosion is a major and costly problem in many industries, including oil, gas, and petrochemicals [1-3]. Corrosion is defined as a process that destroys materials

when they interact with an aggressive environment. This process is characterized by a wide distribution and a variety of conditions and environments in which it occurs [4-6].

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For oil and gas field equipment, the most typical types are general and localized corrosion. General corrosion is understood as a process in which all or some part of the metal surface is subjected to destructive action, while the depth of destruction in some areas may be somewhat greater than in others. Localized corrosion is the most common, which is accompanied by a high rate of metal dissolution in some areas. Corrosion caused by the presence of carbon dioxide in the environment is a critical issue in the petroleum and refining industries during the transportation and processing of oil [7-10]. This occurs due to the pumping of water saturated with carbon dioxide into the wells in order to increase oil recovery and reduce viscosity.

The degree of corrosion on the surface of metals depends on temperature, pressure, pH, and the type of corrosive medium, which are interconnected with each other [11, 12]. The temperature factor plays an important role in the formation of protective films since the rate of chemical and electrochemical reactions at the metal-solution interface changes with increasing temperature. Thus, increasing temperature accelerates the corrosion rate [13]. Moreover, the success of the practical implementation of the protection of oil and gas equipment from corrosion is largely determined by the technical efficiency of the applied method of corrosion control [9]. Today, there are several methods to assess the intensity and nature of the corrosion process. In practice, the following methods are most common: gravimetric (weight loss method), electrical resistance, and linear polarization. In addition, the theoretical determination of corrosion inhibitor activities and modeling of chemical processes is one of the important ways to evaluate the performance of corrosion control under various conditions [14].

Omer *et al.* [15] studied the inhibition activity of naphthalene and tetralin theoretically. For this purpose, the authors analyzed the energy of the highest occupied molecular orbital (E_{HOMO}) and lowest unoccupied molecular orbital (E_{LUMO}). They could determine the role of a donor for each case through the simulation. The authors concluded that naphthalene is more reactive than tetralin, taking on the role of a donor. They demonstrated that the best corrosion inhibition performance can be achieved at higher values of E_{HOMO} and lower values of the energy gap.

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 to an aggressive environment [7, 16-19]. The corrosion inhibition process is associated with a change in the state of the metal surface due to the 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 [20, 21]. Corrosion inhibition occurs through 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 [22, 23]. Furthermore, the adsorption of inhibitors depends on the chemical structure of the inhibitor molecule, electrolyte composition, temperature, types of metals, 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 conducting experiments under the required conditions [24-26].

Prevention of corrosion in the presence of reagents can be associated either with physical adsorption or chemisorption of the inhibitor on the metal surface [27]. Electrostatic attraction between charged hydrophilic groups and the charge of active centers on the metal surface leads to physical adsorption. Most organic inhibitors form a dense barrier film by adsorbing on the metal surface and displacing water molecules from the surface [28]. Meantime, chemisorption is associated with a chemical reaction between the metal surface and the inhibitor components. When the standard free energy of adsorption is less than -20 KJ/mol, the type of adsorption is related to the physical (physisorption), in which inhibition occurs due to electrostatic interaction between the inhibitor molecules and surface. Higher values of the standard free energy of adsorption reflect the chemical adsorption of molecules on the surface [29].

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 [30-33]. 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 [34]. Nitrogen-containing surfactants such as imidazoline and its derivatives have been used successfully to prevent the corrosion process. These organic compounds prevent the invading ions from reaching the metal surface by adsorbing on the surface and forming a rigid mechanical barrier [35, 36]. *He et al.* [16] reported the synergistic inhibition effects of oleic imidazoline and inactive sulfur-containing sulfonate derivatives in HCl solution. They could improve corrosion inhibition performance by determining the optimal mixing ratio of reagents. *Keera et al.* [27] studied imidazoline derivatives for corrosion prevention in the petroleum industry in the carbon steel samples. They observed the high inhibition efficiency at optimal concentrations of the reagents. They mentioned that the adsorption of the imidazoline derivatives followed the Langmuir adsorption isotherm. At the same time, the chemical and electronic structure of benzimidazole derivatives provided inhibitory properties for corrosion prevention and good adsorption on the metal surface. In general, benzimidazole has two preferred sites for the adsorption on metal surfaces: I) nitrogen atom with sp^2 hybrid; II) aromatic ring. *Damej et al.* [5] investigated the inhibition and adsorption of mercaptobenzimidazole on carbon steel samples in a 1 M HCl solution. They noticed that the reagents had good inhibitory properties to control corrosion by adsorbing onto the samples. The Langmuir isotherm model could fully describe the adsorption profile of mercaptobenzimidazole. *Morales-Gil et al.* [28] evaluated the corrosion inhibition efficiency of 2-mercaptobenzimidazole in HCl solution at various concentrations on carbon steel samples. They observed the corrosion protection was highly dependent on concentrations of the inhibitor and acid. Also, 2-mercaptobenzimidazole was adsorbed directly on the carbon steel samples.

Despite numerous studies on the inhibition performance of alone oleic imidazoline or 2-mercaptobenzimidazole, a synergistic inhibition effect between them has not been studied. The ongoing needs of the various industries, including the oil and gas industry, require the study of new and improved existing inhibitor compositions that would be effective at low concentrations. This work, it is aimed to examine the corrosion rate and inhibition performance with and without the use of oleic imidazoline and 2-mercaptobenzimidazole in a 1 M HCl solution for carbon steel samples. For this purpose, weight loss, electrochemical impedance spectroscopy, and potentiodynamic

polarization tests are carried out to determine the optimal concentrations of the reagents. Moreover, the mixtures of oleic imidazoline and 2-mercaptobenzimidazole at different mixing ratios are analyzed to reduce the corrosion rate and increase the inhibition efficiency. The development of an effective mixture of reagents is based on obtaining the maximum synergistic inhibition effect. In addition, it is planned to evaluate the influence of temperature on the inhibition performance of the proposed mixture, oleic imidazoline, and 2-mercaptobenzimidazole. The adsorption isotherm of the corrosion inhibitors on the carbon steel samples is investigated to determine the equilibrium constant and standard free energy. Finally, the inhibition efficiency of the reagents is studied using electrochemical impedance spectroscopy and potentiodynamic polarization tests.

EXPERIMENTAL SECTION

Reagents

To select the components of the mixture of reagents, the possible synergistic effect of a number of known inhibitors in their dual compounds was evaluated in 1 M HCl solution. Among the studied inhibitors, the following reagents could show the greatest performance and synergistic effect: I) oleic imidazoline (OID); II) 2-mercaptobenzimidazole (MBD). Therefore, this paper presents the corrosion inhibition efficiency of these inhibitors and their mixture at different ratios. The chemical structure of these reagents is shown in Fig. 1. CAS number of 2-mercaptobenzimidazole was 583-39-1 (Merck). Moreover, oleic imidazoline was prepared by the reaction of diethylenetriamine and oleic acid in the laboratory. For this purpose, 16 mL of oleic acid was added to a three-necked round-bottom flask equipped with a thermocouple, a magnetic stirrer, and a drip funnel. The solution was heated to 60 °C and then 11 mL of diethylenetriamine was added to it. The mixture was then heated to 160 °C and kept at this temperature for 4 h to accumulate water formed as a result of the reaction. After that, the temperature was raised to 225 °C and the mixture was kept at this temperature for 1 hr. During this time, a stream of nitrogen was applied to the solution and the stirring speed was increased. In the next step, the temperature of the solution was maintained at 225 °C for another 2 h. It was then cooled to room temperature and purified by extraction with water and chloroform.

Table 1: The composition of the carbon steel samples used in the experiments.

Composition	C	Si	Mn	P	S	Fe
wt. %	0.15	0.30	0.65	0.02	0.10	98.78

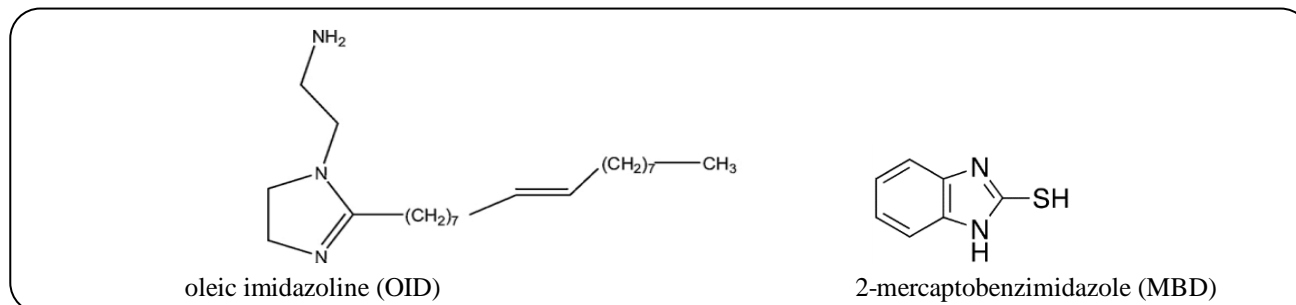


Fig. 1: The chemical structure of the corrosion inhibitors.

Weight loss method for determining the corrosion rate and evaluating the performance of reagents

To assess the performance of the corrosion inhibitors, carbon steel samples (20 mm - length × 10 mm - width × 2 mm - thickness) were used. As shown in Table 1, 98.8% of the carbon steel samples used were iron. In the tests, the samples were immersed in the 50 mL HCl solution (1 M) both in the presence and absence of the corrosion inhibitors. The mass of the samples was measured every 30 min for 14 hr. Also, the inhibitors were used at various concentrations to determine the optimal values. The tests were completed at 333.15 K. Each test was repeated twice, and the average weight of the carbon steel samples was used. After determining the optimal concentrations of the reagents, the tests were carried out in the range of 298.15-373.15 K to evaluate the effect of temperature on the corrosion inhibition performance.

The corrosion rate of the samples in the solution, the inhibition efficiency of the reagents, and surface coverage were determined as follows:

$$R_c = \frac{87.6\Delta m}{Atp} \quad (1)$$

$$CIE = \frac{R_{c1} - R_{c2}}{R_{c1}} \times 100 \quad (2)$$

$$\theta = \frac{CEI}{100} \quad (3)$$

Where R_c is the corrosion rate (mm/y); Δm is the average weight loss of the samples (mg); A is the surface area of the used samples (mm²); t is the experiment time (hr); ρ is the density of the samples (gr/cm³); CIE is

the corrosion inhibition efficiency (%); R_{c1} is the corrosion rate in the absence of inhibitor (mm/y); R_{c2} is the corrosion rate in the presence of inhibitor (mm/y); θ is the surface coverage (dimensionless).

The effect of the mixture of reagents

To increase the corrosion inhibition performance, reagents of OID and MBD were used as a mixture at the following ratios: 0:100, 10:90, 20:80, 25:75, 30:70, 40:60, 50:50, 60:40, 70:30, 75:25, 80:20, 90:10, 100:0. The mixture of inhibitors was added to the HCl solution at different concentrations. The inhibition efficiency of the mixtures was determined using Equation 2 for 14 h. At the same time, the tests were performed at 75 °C. Moreover, the synergistic inhibition effect was evaluated by the following formula [37]:

$$\text{Synergy} = \frac{CIE_m - \text{Average}(CIE_{OID}, CIE_{MBD})}{\text{Average}(CIE_{OID}, CIE_{MBD})} \times 100 \quad (4)$$

Where CIE_m is the corrosion inhibition efficiency of a mixture of reagents (%); CIE_{OID} is the corrosion inhibition efficiency of 100% oleic imidazoline (%); CIE_{MBD} is the corrosion inhibition efficiency of 100% 2-mercaptobenzimidazole (%).

Adsorption isotherm of the corrosion inhibitors

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 between the values of the surface coverage (θ) and the inhibitor concentration (C_{inh}). The Langmuir isotherm, in contrast to other isotherms,

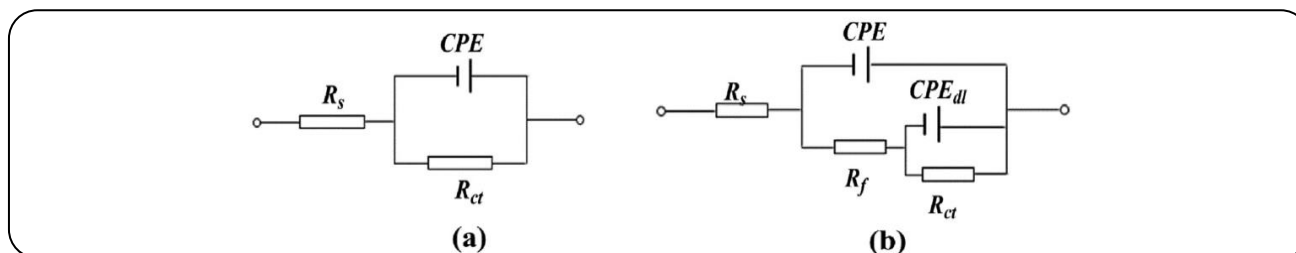


Fig. 2: The equivalent circuits applied to fit data in EIS tests in the absence (a) and presence of corrosion inhibitors [38].

most fully described the adsorption behavior of the molecules of the studied inhibitors. According to this isotherm, θ is related to C_{inh} by the following equation [38]:

$$\frac{C_{inh}}{\theta} = C_{inh} + \frac{1}{K_{ads}} \quad (5)$$

Where C_{inh} is agent concentration (M); θ is surface coverage, K_{ads} is the equilibrium constant of the adsorption process of the inhibitor.

According to Equation (5), 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]:

$$\Delta G^{\circ}_{ads} = -RT \ln(55.5K_{ads}) \quad (6)$$

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

Electrochemical Impedance Spectroscopy (EIS) and potentiodynamic polarization tests

The electrochemical methods, in contrast to the weight loss method, make it possible to evaluate not only the mass loss of the metal samples but also the effect of inhibitors on the kinetics of the cathode and anode processes. EIS tests were conducted at Open Circuit Potential (OCP) in 1 M HCl solution in order to analyze the corrosion inhibition efficiency and obtain Nyquist, Bode, and phase angle plots. For this purpose, CorrTest potentiostat was used. Three electrodes were employed in the setup: reference, counter, working. The tests were performed in the presence and absence of the reagents. The corrosion inhibitors were applied at various concentrations at 60 °C. Fig. 2 demonstrates the equivalent circuits applied to fit data in EIS tests in the absence and presence of corrosion inhibitors. Where, CPE is the constant phase element; CPE_{dl} is the CPE describing the double-layer capacitance ($\mu F/cm$);

R_s , R_f and R_{ct} are the solution, film, and charge transfer resistances, respectively (ohm.cm^2). In this case, the corrosion inhibition efficiency was determined as follows:

$$CIE = \frac{R_{ct2} - R_{ct1}}{R_{ct2}} \times 100 \quad (7)$$

Where R_{ct1} and R_{ct2} are the charge transfer resistances in the absence and presence of inhibitors in the solution, respectively, (ohm.cm^2).

Moreover, the potentiodynamic polarization tests were carried out in a wide potential range using a Saturated Calomel Electrode (SCE) as a reference electrode at the OCP. These tests were completed to examine the inhibition performance of the mentioned corrosion inhibitors and their mixture. For this purpose, the following potentiodynamic polarization parameters were determined in the presence and absence of the inhibitors at 60 °C: E_{corr} - corrosion potential (mV) and i_{corr} - corrosion current density ($\mu A/cm^2$). The corrosion inhibition efficiency was calculated as follows:

$$CIE = \frac{i_{corr1} - i_{corr2}}{i_{corr1}} \times 100 \quad (8)$$

Where i_{corr1} and i_{corr2} are the corrosion current densities in the absence and presence of inhibitors in the solution, respectively, ($\mu A/cm^2$).

RESULTS AND DISCUSSION

The corrosion inhibition performance of the reagents by weight loss method

An important tool for preventing corrosion is the use of compounds with high efficiency, low cost, and low toxicity [21]. Fig. 3 presents the results of corrosion rate and inhibition efficiency of OID at 60 °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 hr immersion of the carbon steel samples in the HCl solution, the corrosion rate

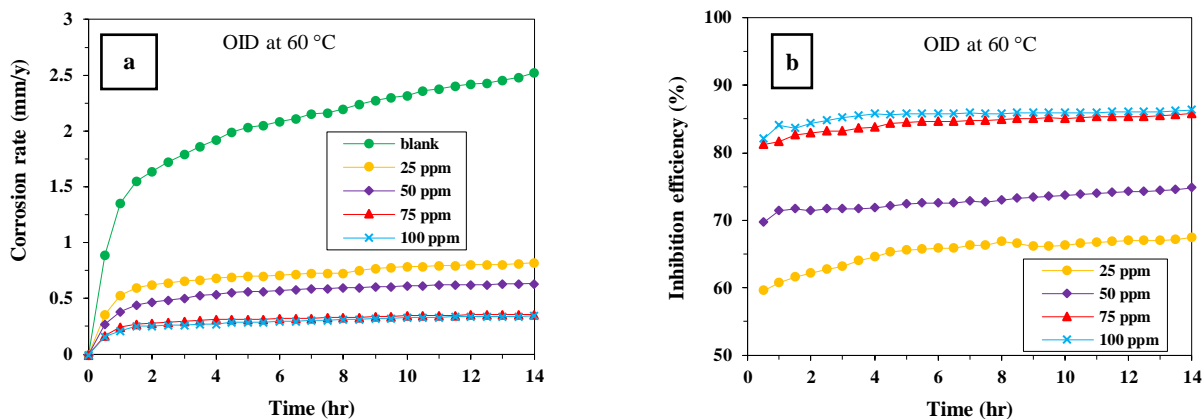


Fig. 3: The changes in the corrosion rate (a) and inhibition efficiency (b) of oleic imidazoline (OID) depending on the concentration and test time at a constant temperature of 60 °C in the weight loss method.

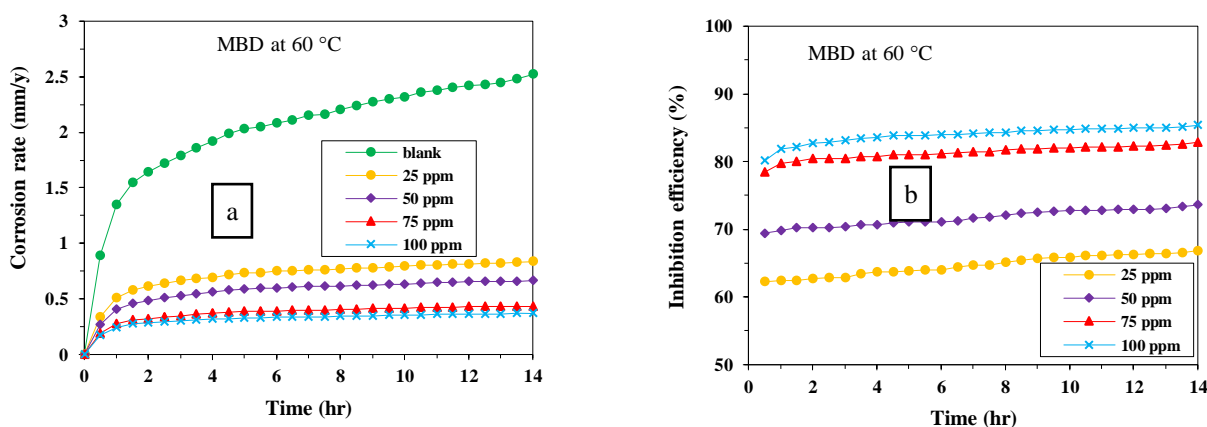


Fig. 4: The changes in the corrosion rate (a) and inhibition efficiency (b) of 2-mercaptobenzimidazole (MBD) depending on the concentration and test time at a constant temperature of 60 °C in the weight loss method.

was higher than 2.5 mm/y in the absence of the inhibitor. The addition of OID 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 by increasing the concentration of the inhibitor. The optimal concentration of OID was obtained 75 ppm, as the corrosion rate and inhibition performance were not noticeably changed at higher concentrations. As shown in Fig. 3, the corrosion rate and inhibition efficiency after 14 h at 75 ppm were 0.36 mm/y and 85.8%, respectively. This corrosion rate is high for oilfield conditions.

Fig. 4 shows the weight loss of carbon steel samples in a 1 M HCl solution in the absence and presence of 2-mercaptobenzimidazole at various concentrations. The tests were performed at 60 °C. In this case, the corrosion protection efficiency was calculated using Equation (2). In the presence of MBD at 75 ppm and 100 ppm, the minimal loss of the mass was observed at the investigated temperature. As the inhibitor concentration was increased, the corrosion inhibition efficiency and the degree of surface coverage were increased. In addition, with an increase in the concentration, as a result of the interaction of the inhibitor molecules with the surface, the density of the adsorbed layer was increased. As illustrated in Fig. 4, the optimal concentration of MBD was obtained 75 ppm. At this concentration, the corrosion rate and inhibition

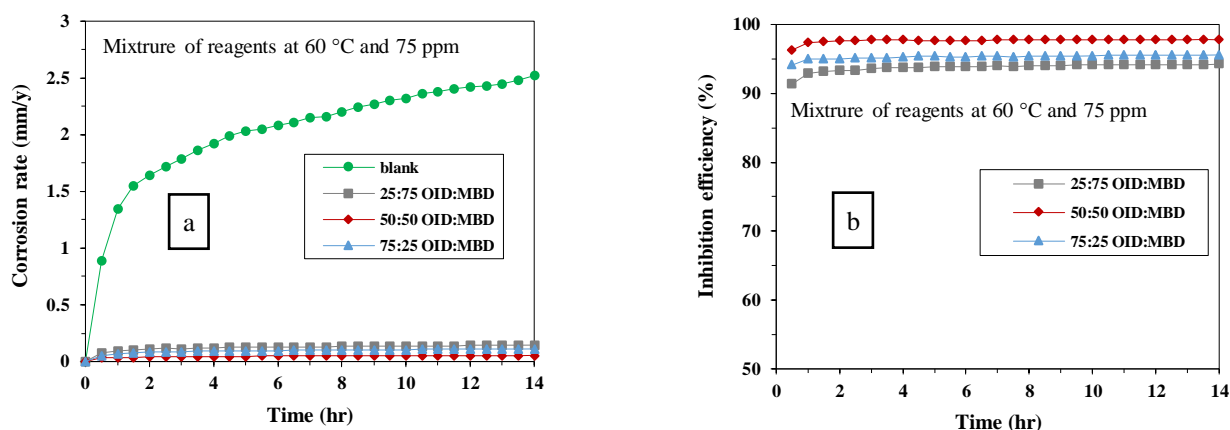


Fig. 5: The changes in the corrosion rate (a) and inhibition efficiency (b) of the mixture of OID and MBD depending on the mixing ratio and test time at a concentration of 75 ppm and 60 °C in the weight loss method.

efficiency were 0.43 mm/y and 82.9%, respectively. The inhibition performance of OID and MBD was insufficient for use in real cases. Thus, the corrosion inhibition performance should be improved through the development of new reagents or the proposal of mixtures with a positive synergistic inhibition effect. Therefore, corrosion protection was investigated using mixtures of these two inhibitors at various mixing ratios (presented in the next section).

The corrosion inhibitor after mixing of the reagents

To reduce the corrosion rate and increase the inhibition performance, OID and MBD were used as a mixture at various mixing ratios. To this end, the weight loss method was applied in order to determine the corrosion rate and inhibition efficiency at 60 °C. Fig. 5 illustrates the results of corrosion inhibition in the presence of the mixtures of OID and MBD at ratios of 25:75, 50:50, and 75:25. The mixtures were used at 75 ppm, since the optimal concentrations of both reagents were observed at this value. The best results in preventing corrosion were found at a ratio of 1:1 OID and MBD. At this mixing ratio, the corrosion rate and inhibition efficiency were obtained 0.055 mm/y and 97.8%, respectively. Thus, the inhibitory ability of the mixture of reagents was higher than either OID or MBD alone, by more than 12%. Furthermore, the synergistic effect of the mixtures in preventing corrosion is depicted in Fig. 6 (data in Table 2). When a synergistic effect is observed among the components of a mixture, its inhibition performance is enhanced compared to each individual component [37-41]. The greatest synergistic

inhibition effect was 15.9%, which occurred at a mixing ratio of 1:1 (50:50) of OID:MBD. Therefore, the mixture of oleic imidazoline and 2-mercaptobenzimidazole at a ratio of 1:1 at 75 ppm is used in further experiments. The high value of corrosion inhibition efficiency, close to 100%, indicates an almost complete coverage of the carbon steel surface with adsorbed molecules of the reagents. In this case, the space between adsorbed inhibitor molecules on the surface can be less than the area of the molecules, which increases the efficiency of corrosion inhibition. Moreover, Table 3 presents the data from the weight loss method for carbon steel samples without (blank case) and with the corrosion inhibitors at different concentrations after 14 hr immersion in 1 M HCl at 60 °C. As can be seen in the table, the highest corrosion performance refers to the mixture of MID and MBD. The main reason for the high efficiency of the mixture is the creation of a dense adsorption layer on the surface of the carbon steel samples. In this case, various inhibitory effects of the components can also be expected simultaneously. The optimal concentration of the mixture of the reagents was 75 ppm.

Effect of temperature on the corrosion inhibition efficiency

Along with the assessment of the protective effect of inhibitors, the characteristic of their technological properties is no less important. Technological properties are evaluated in relation to specific operating conditions. Temperature is one of the main parameters affecting the corrosion inhibition process [18]. Fig. 7 presents the influence of temperature on the inhibition performance

Table 2: Data of the corrosion inhibition efficiency and synergistic inhibition effect at 75 ppm and 60 °C in the weight loss method.

Test number	OID content in the mixture (%)	MBD content in the mixture (%)	CIE by weight loss (%)	Synergy by Eq. 4 (%)
1	0	100	82.9	---
2	10	90	86.6	2.7
3	20	80	90.2	6.9
4	25	75	94.2	11.7
5	30	70	94.8	12.4
6	40	60	96.2	14.0
7	<u>50</u>	<u>50</u>	<u>97.8</u>	<u>15.9</u>
8	60	40	96.4	14.3
9	70	30	96.1	13.9
10	75	25	95.6	13.3
11	80	20	92.8	10.0
12	90	10	88.9	5.4
13	100	0	85.8	---

Table 3: Results of weight loss tests for carbon steel samples in 1 M HCl in the absence and presence of the corrosion inhibitors at various concentrations at 60 °C with an immersion time of 14 hr.

Inhibitor	Concentration (ppm)	Δm (mgr)	A (cm ²)	R _c (mm/y) by Eq. 1	CIE (%) by Eq. 2
blank	0	1644	520	2.52	---
OID	50	711	520	0.635	74.77
MBD	50	744	520	0.666	73.59
50:50 OID:MBD	50	240	520	0.210	91.67
OID	75	400	520	0.357	85.83
MBD	75	483	520	0.432	82.86
50:50 OID:MBD	75	62	520	0.055	97.82
OID	100	385	520	0.345	86.32
MBD	100	413	520	0.369	85.36
50:50 OID:MBD	100	59	520	0.053	97.90

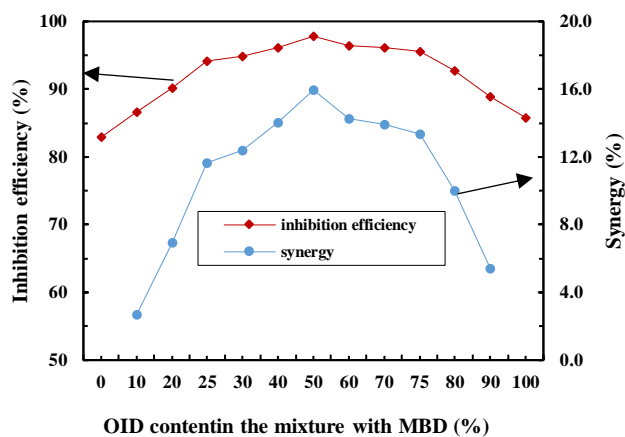


Fig. 6: Corrosion inhibition efficiency and synergy at various mixing ratios of OID and MBD at 75 ppm and 60 °C in the weight loss method.

of the studied reagents. In the present case, the inhibitors were used at a concentration of 75 ppm. The mixture of reagents was applied at a ratio of 1:1. It was observed that the corrosion inhibition efficiency was not significantly decreased, as the temperature was increased from 298.15 to 373.15 K. The inhibition performance of OID, MBD, and the mixture of reagents at 373.15 K was 81.7, 79.0, and 93.7%, respectively. A slight decrease in the inhibition efficiency by increasing temperature is associated with the chemical transformations of inhibitor molecules and their desorption from the surface of the carbon steel samples.

Adsorption isotherm of the corrosion inhibitors

As shown in Fig. 8, 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, OID, MBD, and the mixture of these inhibitors at a ratio of 1:1 were tested at 60 °C. The coefficient of determination (R^2) was practically equal to unity for all reagents. The high values of the coefficients of the determination indicate 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 Eq. (5). The results are presented in Table 4. High K_{ads} values demonstrate that the inhibitor molecules have strong adsorption capacity on the surface of the carbon steel samples. The mixture of reagents had

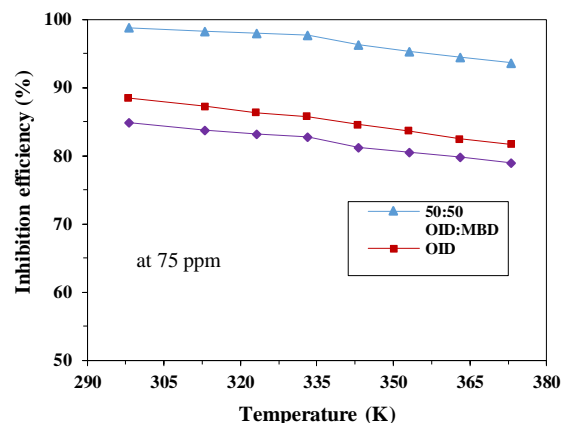


Fig. 7: The dependence of corrosion inhibition efficiency on the temperature in the presence of inhibitors at 75 ppm after 14 h by the weight loss method.

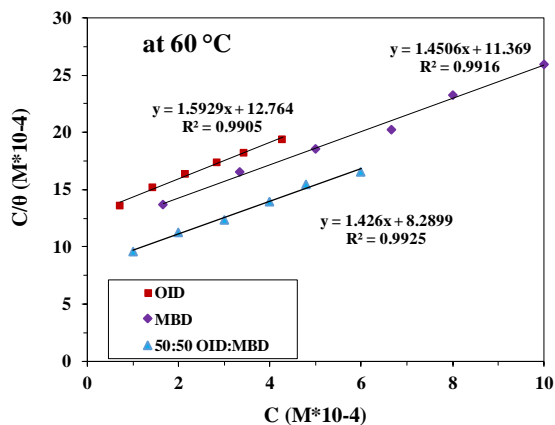
the highest K_{ads} value. In addition, the standard free energy of adsorption (ΔG°_{ads}) was calculated using Equation 6 for all inhibitors. The results are also illustrated in Table 4. Negative values of ΔG°_{ads} are consistent with the spontaneity of the adsorption process and the stability of the adsorbed layer on the surface of carbon steel samples. The values of ΔG°_{ads} for the corrosion inhibitors ranged from -31.36 to -32.62 KJ/mol, which indicates that the adsorption mechanism on the surface of carbon steel in 1 M HCl solution corresponds to both physisorption (electrostatic), and chemisorption (charge sharing) features, since the values of ΔG°_{ads} were between 20 and 40 KJ/mol [42, 43]. Also, the chemical bond between the mixture of reagents and iron was stronger than the bond between OID or MBD and iron, since the absolute value of the standard free energy of adsorption and the equilibrium constant of the adsorption process of the proposed mixture was greater than only OID or MBD. Thus, this indicates the presence of strong adsorption between the molecules of the used mixture and the surface of the carbon steel samples. This behavior is associated with a higher electron density at the adsorption site of the mixture of reagents.

EIS and polarization tests

EIS tests were completed in 1 M HCl solution in the presence and absence of corrosion inhibitors at 60 °C. The reagents were used at 75 ppm. Nyquist plots obtained from the EIS tests are shown in Fig. 9. In the absence of inhibitors, the Nyquist plot is a semicircle with a small diameter, which may be associated with charge transfer

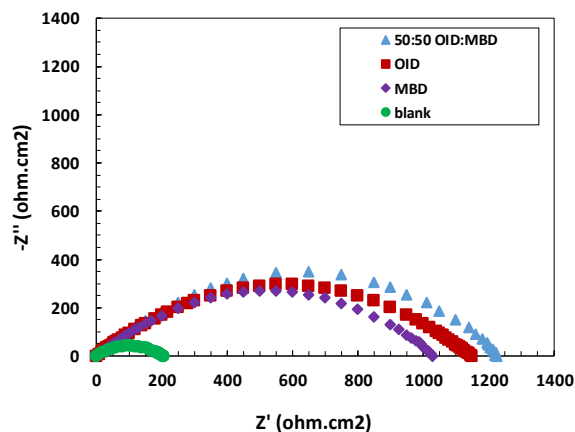
Table 4: The adsorption parameters of the corrosion inhibitors determined on the Langmuir isotherm.

Inhibitor	R ²	K _{ads} (M)	ΔG° _{ads} (KJ/mol)
OID	0.9905	783.5	-31.36
MBD	0.9916	879.6	-31.70
50:50 OID:MBD	0.9925	1206.3	-32.62

**Fig. 8: Langmuir isotherm for the adsorption of corrosion inhibitors on the surface of samples at 60 °C.**

reactions occurring on the surface of carbon steel samples in the solution. This phenomenon is related to the dual-layer capacity and charge transfer resistance. Thus, the diameter of semicircles can be regarded as a measure of the charge transfer resistance. The lower the charge transfer resistance (smaller diameter of the semicircles), the higher the corrosion rate. As illustrated in Fig. 9, the diameter of the semicircles is larger in the presence of each corrosion inhibitor than in that case where there is no inhibitor in the solution. This fact refers to the inhibition ability of the reagents. The largest diameter of semicircles was observed for the mixture of oleic imidazoline and 2-mercaptobenzimidazole at the ratio of 1:1. The results demonstrate that the mixture of reagents increased the charge transfer resistance by increasing the degree of protection of carbon steel samples from corrosion and adsorption on the surface. Therefore, the suggested mixture of reagents had a better inhibition performance for preventing corrosion in comparison with only OID or MBD.

The Bode and phase angle plots for the used carbon steel samples in the absence and presence of the corrosion inhibitors are presented in Fig. 10. The inhibitors were added to the 1 M HCl solution at 75 ppm and the tests

**Fig. 9: Nyquist plots for carbon steel samples in 1 M HCl solution in the presence and absence of corrosion inhibitors at 60 °C.**

were completed at 60 °C. In the Bode plots, the highest impedance values (absolute) at lower frequency depict a high corrosion inhibition efficiency in the studied solution [38]. As presented in Fig. 10a, the impedance values were increased in the presence of inhibitors compared to a blank case. It is related to the formation of a protective layer on the surface of the carbon steel samples, which prevents the corrosion process. The formation of a protective layer on the surface is provided by the different charges of inhibitor particles and their ability to form strong chemical bonds with metal cations. More details on the inhibition mechanism of the used inhibitors are explained in the next section. Among the used reagents, the mixture of OID and MBD at a ratio of 50:50 had the highest impedance values at the lowest frequencies. Thus, these results also confirm the better performance of the mixture of reagents than individually. In this case, the best ability to inhibit corrosion is provided at more negative values of the phase angles [38]. As illustrated in Fig. 10b, the highest amount of phase angles (absolute) occurred in the presence of a mixture of OID and MBD. Thus, the results of the phase angle plots show that the proposed mixture had the best corrosion performance for carbon steel samples in a 1 M HCl solution.

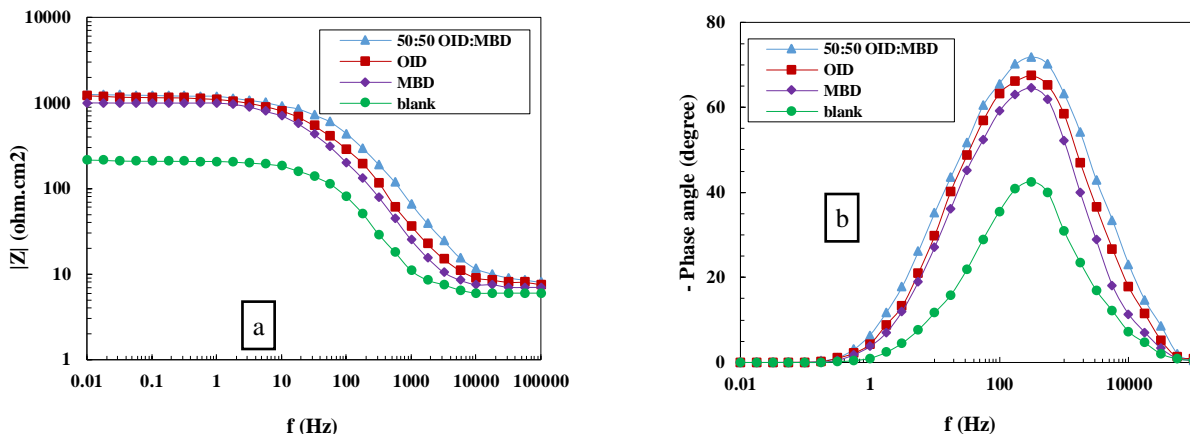


Fig. 10: The Bode (a) and phase angle (b) plots without and with the use of corrosion inhibitors at 75 ppm and 60 °C in 1 M HCl solution.

Electrochemical parameters of impedance spectroscopy and potentiodynamic polarization tests in the absence and presence of corrosion inhibitors at different concentrations are depicted in Table 5. As shown in the table, the corrosion inhibition efficiency was increased by increasing the inhibitor concentration in both tests. The charge transfer resistance (R_{ct}) was increased with an increase in the concentration of the inhibitors, indicating that they react with the surface of the carbon steel samples to prevent corrosion. At higher values of R_{ct} , the level of corrosion protection was high. Furthermore, the corrosion potential (E_{corr}) was changed by increasing the concentrations of the reagents, which indicates a mixed-type inhibitor. Thus, the studied reagents affected both the cathodic and anodic reactions. As presented in Table 5, the corrosion current density was significantly reduced in the presence of the inhibitors compared to the blank case. This reduction is related to the adsorption of the reagents on the surface of the samples. As shown in the table, the proposed mixture of reagents had the highest corrosion inhibition efficiency in EIS and potentiodynamic polarization tests such as the weight loss method. Therefore, the surface of carbon steel samples immersed in 1 M HCl solution has the least corrosion reaction products due to the anti-corrosive properties of the studied reagents in the mixture. The obtained results confirmed that the optimal concentration of the mixture is 75 ppm.

Corrosion inhibition mechanism of the studied reagents

The inhibitory action of the used reagents was associated with the formation of a protective layer through the

adsorption of reagents on the surface of carbon steel samples. All conducted experiments showed the high performance of the mixture of reagents for corrosion inhibition at various temperatures at a low concentration compared to a single reagent. The adsorption isotherm of the used inhibitors obeyed the Langmuir type. The adsorption behavior is characterized by the interaction of inhibitor molecules and the surface of the samples. MBD adsorption occurs through exocyclic S and N atoms. Based on the previous works [44–47], the S and N atoms in the OID and MBD structure participate in surface bonds. Thus, these are utilized as active adsorption sites for the enhancement of corrosion inhibition performance. In addition, the presence of C=N (in both OID and MBD) and C=S (in MBD) groups can lead to the formation of π -bonds, providing high corrosion inhibition characteristics [44, 47, 49]. The adsorption and inhibition mechanism of OID and MBD on the surface of carbon steel samples are presented in Fig. 11. It should be noted that the presence of π -electrons and N heteroatoms makes the adsorption of the inhibitors on the surface of the carbon steel samples due to lone pairs. Heteroatoms and π -electrons can support electron donation to the empty d-orbital of iron atoms, leading to greater adsorption of the inhibitor on the surface of the carbon steel samples and better corrosion inhibition performance. As mentioned above, the adsorption of the reagents on the surface of the samples was carried out according to both physisorption (electrostatic) and chemisorption features. Therefore, in addition to chemisorption, the adsorption of the studied inhibitors also occurred by electrostatic attractions, which could more

Table 5: Electrochemical parameters of impedance spectroscopy and potentiodynamic polarization tests for carbon steel samples in 1 M HCl in the absence and presence of the corrosion inhibitors at different concentrations at 60 °C.

Inhibitor	Concentration (ppm)	R_{ct} (ohm.cm ²)	CIE (%) by Eq. 7	E_{corr} (mV)	i_{corr} (μA/cm ²)	CIE (%) by Eq. 8
blank	0	52	---	-441	724	---
OID	50	233	77.68	-430	142	80.39
MBD	50	217	76.04	-432	154	78.73
50:50 OID:MBD	50	685	92.41	-423	47	93.51
OID	75	342	84.80	-422	87	87.98
MBD	75	305	82.95	-425	106	85.36
50:50 OID:MBD	75	1358	96.17	-412	12	98.34
OID	100	397	86.90	-414	74	89.78
MBD	100	354	85.31	-417	92	87.29
50:50 OID:MBD	100	1486	96.50	-402	11	98.48

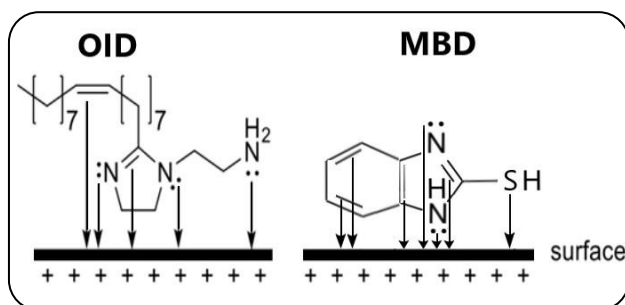


Fig. 11: The adsorption and inhibition mechanism of OID and MBD on the surface of the samples [45, 50].

effectively prevent corrosion in the aggressive medium. Despite the discussed possible mechanisms, an in-depth study on the mechanisms of the used inhibitors will be performed in future works conducting more advanced tests such as Scanning Electron Microscopy (SEM) and X-ray Photoelectron Spectroscopy (XPS), Molecular Dynamics (MD) simulations, Monte Carlo, and quantum mechanics.

CONCLUSIONS

In this work, the corrosion inhibition performance was analyzed by conducting various experiments for carbon steel samples in a 1 M HCl solution. For this purpose, we used a set of well-known corrosion inhibitors in order to develop a mixture with enhanced efficiency, showing a positive synergistic effect. Among the studied reagents, oleic imidazoline and 2-mercaptobenzimidazole had the highest synergistic effect. Thus, the inhibition performance of the mixtures of these inhibitors was

examined. The results of weight loss tests showed that the corrosion rate and inhibition efficiency of OID and MBD at an optimal concentration of 75 ppm were 0.36 mm/y, 0.43 mm/y, 85.8%, 82.9%, respectively. These values are insufficient to prevent corrosion in some industries, including the petroleum industry. Moreover, the mixtures of OID and MBD indicated better inhibition performance than a single inhibitor. The highest inhibition efficiency of the mixture of reagents was 97.8% at a mixing ratio of 1:1. It was related to the appearance of a positive synergistic inhibition effect among the used components. In this case, the synergy between OID and MBD at a ratio of 1:1 was 15.9%. Thus, this mixture (50:50 OID:MBD) was used in further experiments. In addition, with an increase in the temperature from 298.15 to 373.15 K, the inhibition efficiency of the corrosion inhibitors was not significantly reduced, which was due to the complete adsorption of the reagents on the surface of the carbon steel samples. Also, the experimental results showed that the adsorption of the inhibitors on the surface of the samples followed the Langmuir isotherm. In this case, the coefficient of determination for the studied reagent was almost 1. In addition, the equilibrium constant for the adsorption process of the proposed mixture of reagents was higher than either OID or MBD alone. The values of ΔG_{ads}° indicated that the adsorption mechanism of the studied reagents corresponds to both physisorption, and chemisorption features. Furthermore, the Nyquist, Bode, and phase angles plots for carbon steel samples in the presence and absence of corrosion inhibitors

were determined at 60 °C. The results confirmed a superior inhibition performance of the proposed mixture of reagents for corrosion control compared to OID or MBD alone. The used reagents behaved as a mixed-type corrosion inhibitor and affected both the cathodic and anodic reactions. Finally, the high corrosion inhibition efficiency of the developed mixture was associated with the presence of C=N and C=S groups in the structure of its components and the simultaneous manifestation of several inhibitory effects.

In the end, although our results from the weight loss method, EIS, and potentiodynamic polarization tests show high corrosion efficiency of the mixture of reagents under various conditions, a deeper assessment at the microscopic level of the mechanisms and interactions of inhibitors with the surface of the samples is still essential. This is a topic for our future work that can be analyzed by carrying out additional tests such as SEM and XPS, which can show the formation of an adsorbed inhibitor layer on the surface of carbon steel samples and its protective characteristics.

Nomenclature

A	Surface area of the used samples, mm ²
CIE	Corrosion inhibition efficiency, %
C _{inh}	Reagent concentration, M
CPE	Constant phase element
CPE _{dl}	CPE describes the double-layer capacitance, μF/cm ²
E _{corr}	Corrosion potential, mV
EIS	Electrochemical impedance spectroscopy
E _{HOMO}	Energy of the highest-occupied molecular orbital, eV
E _{LUMO}	Energy of the lowest unoccupied molecular orbital, eV
i _{corr}	Corrosion current density, μA/cm ²
K _{ads}	Equilibrium constant of the adsorption process
MBD	2-mercaptobenzimidazole
MD	Molecular dynamics
OCP	Open circuit potential
OID	Oleic imidazoline
R	Universal gas constant
R _c	Corrosion rate, mm/y
R _{ct}	Charge transfer resistance, ohm.cm ²
R _f	Film resistance, ohm.cm ²
R _s	Solution resistance, ohm.cm ²
R ²	Coefficients of determination
SCE	Saturated calomel electrode

SEM	Scanning electron microscopy
t	Experiment time, hr
T	Temperature, K
XPS	X-ray photoelectron spectroscopy
ΔG [°] _{ads}	Standard free energy of adsorption, J/mol
Δm	Weight loss of the samples, mg
θ	Surface coverage, dimensionless
ρ	Density of the samples, g/cm ³

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