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2-(2,4-Dimethoxybenzylidene)-N-Phenylhydrazinecarbothioamide as an Efficient Corrosion Inhibitor for Mild Steel in Acidic Environment

R. D. Salim[*1], N. Betti[2], M. Hanoon[3], A. A. Al-Amiery [*4, 5]

[1.] Electrical Engineering Technical College, Middle Technical University, P.O. Box: 1001, Baghdad, Iraq

[2.] Materials Engineering Department, University of Technology, P.O. Box: 1001, Baghdad, Iraq

[3.] Production Engineering and Metallurgy, University of Technology, P.O. Box: 1001, Baghdad, Iraq

[4.] Department of Production Engineering and Metallurgy, University of Technology, P.O. Box: 1001, Baghdad, Iraq

[5.] Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, Bangi, Selangor 43600, Malaysia

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ABSTRACT

In this work, a new Schiff base, namely 2-(2,4-dimethoxybenzylidene)-N-phenylhydrazinecarbothioamide (DP), was synthesized and fully characterized by some spectroscopical techniques (Fourier Transform Infrared (FT-IR), and Nuclear Magnetic Resonance (^{1}H-NMR and ^{13}C-NMR) in addition to micro elemental analysis-CHN. The newly synthesized corrosion inhibitor was evaluated for its corrosion inhibition performance on mild steel coupons in 1 M hydrochloric acid solution by using gravimetric techniques. The experimental findings of weight loss measurements revealed that the inhibition efficiency increased with the DP concentration and reached a maximum value of 94.8% at the 0.005 M concentration but decreased with reducing temperature (at temperatures ranging from 303 to 333 K). Moreover, the significant inhibition efficiency and the value of ΔG^{o} indicated that DP participates in Chemisorption and Physisorption on the mild steel surface. The adsorption process of the synthesized inhibitor on a mild steel surface follows Langmuir adsorption isotherm. The uninhibited and inhibited surface morphology of the mild steel coupons was investigated using scanning electron microscopy (SEM). Prog. Color Colorants Coat. 15 (2022), 45-52© Institute for Color Science and Technology.

1. Introduction

The corrosion damages generate high inspection, replacement, and repair costs; however, besides those constituting public risks, the importance of the development of new substances behaving similarly to the inhibitors of corrosion, particularly in the acidic media [1]. Using organic molecules as corrosion inhibitors is an efficient method for protecting corrosions, and it has become increasingly popular [2]. The available data have shown that the organic inhibitors act through adsorption on the metal's surface and the protective film's formation [3]. It was exhibited that the organic compounds that contain the heteroatoms with a high electron density, like nitrogen, phosphorus, oxygen, and sulfur, in addition to the ones that have several bonds that are viewed as centers of adsorption, are efficient as inhibitors of corrosion [4]. Synthesized Schiff-bases showed more than 90% efficiency of the inhibition at lower concentrations than others provided in the literature [5]. Lately, the Schiff bases' compounds

*Corresponding author: * dr.ahmed1975@gmail.com;
100173@uotechnology.edu.iq

were of interest to obtain sufficient inhibitors of corrosion [6], since they are providing considerably higher inhibition compared with the corresponding aldehydes and amines [7]. The existence of (C=N) group in the Schiff base molecules results in enhancing their ability for the efficiency of adsorption and corrosion inhibition [8]. This inhibitor 2"-(2,4-dimethoxy-benzylidene)-N-phenylhydrazinecarbothioamide" was chosen because its synthesis technique is safe and straightforward. It is synthesized from thiosemicarbazide that is cheap, and used to manufacture many drugs and medical materials.

Continuing previous corrosion investigation [9-18], this study reports anti-corrosive Schiff base behavior for the mild steel in the HCl solution. Therefore, scanning electron microscopy (SEM) and weight loss approaches have been utilized.

2. Experimental

Chemicals (Analytical grade) were utilized to synthesize the corrosion inhibitor and were purchased from Merck and Sigma Aldrich and utilized without further purification. The composition of the mild steel specimen is depicted in Table 1. The coupon dimensions 4 × 1.5 × 0.1 cm were utilized for weight loss measurements. The coupons were polished with emery paper and washed with double distilled water, dried and rinse with acetone and dry with an oven.

2.1. Synthesis of the corrosion inhibitor

The inhibitor 2-(2,4-dimethoxybenzylidene)-N-phenylhydrazinecarbothioamide (DP), as shown in Figure 1, was synthesized by refluxing of 2,4-dimethoxybenzaldehyde (0.01 mm) and N-phenylhydrazinecarbothioamide (0.01 mm) using ethyl alcohol as the solvent for 12 hours. The mixture was filtered, recrystallized from ethyl alcohol, and dried.

2.2. Characterization of the corrosion inhibitor

Yield 65%, white Solid, m.p. 193–196°C., FT-IR (cm^{-1}); 3305, and 3249 (amino groups), 3089 (C-H aromatic), 2949 (C-H aliphatic), 1621 (C=N), and 1547, (C=C); ^{1}H NMR (400 MHz, DMSO-d6) d(ppm): 3.29 (s, 3H, methoxyide), 3.83 (s, 3H, methoxyide), 8.36(s, 1H, amine), 6.91–7.07 (aromatic protons); ^{13}C NMR (400 MHz, DMSO-d6) d(ppm):147.7, 55.9, 100.6, 106.7, 112.2, 126.4, 127.2, 128.3, 130.1, 138,8, 159.5, 163.1, 18479.1; CHN elemental Analysis: C, 60.93 (61.03); H, 5.43 (5.47); N, 13.32 (13.83).

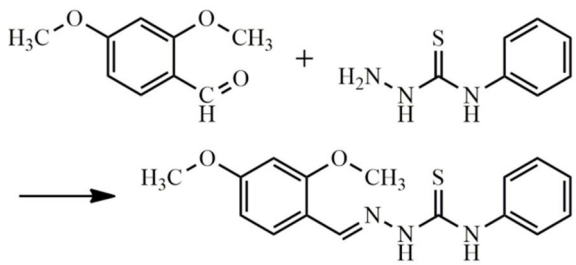

Figure 1: Synthetic route of the DP.

Table 1: Compersion between the investigated inhibitor and other published inhibitors.

No.	Inhibitor	IE%
1	4-(Benzoimidazole-2-yl)pyridine	91.16
2	4-Dimethylamino-benzylidene)-[1, 3, 4]thiadiazol-2-yl-amine	91
3	4-hydroxybenzylideneaminomethyl-5-ethyl-1,3,4-thiadiazol	90
4	2-Methyl-4H-benzo[d][1,3]oxazin-4-one	65
5	3-amino-2-methylquinazolin-4(3H)-one	89
6	3-((5-(3,5-dinitrophenyl)-1,3,4-thiadiazol-2-yl)imino)indolin-2-one	90.7
7	3-nitro-5-(2-amino-1,3,4-thiadiazolyl)nitrobenzene	92.3
8	3-((4-(dimethylamino)benzylidene)amino)-2-methylquinazolin-4(3H)-one	92
9	4-((5,5-dimethyl-3-oxocyclohexenyl)amino)benzenesulfonamide	93
10	2-((2-hydroxy-5-methoxybenzylidene)amino)pyridine	90

2.3. Weight loss measurements

Coupons of mild steel were immersed in a 1.0 M hydrochloric acid solution with various inhibitor concentrations (0.001, 0.002, 0.003, 0.004, and 0.005 M) used for periods of 1, 5, 10 and 24 h in a thermostatically controlled water bath at 303 K under aerated conditions. Weight loss measurements were also performed for various temperatures (303, 313, 323, and 333 K) and different inhibitor concentrations for five hours as immersion time [20-22]. The weight loss measurements were performed in triplicate, and the average was reported.

2.4. Scanning electron microscopy

Screening of surface specimens in the absence and presence of DP at the concentration of 0.005 M which was immersed for 5 h at 303 K, was studied by scanning electron microscopy (Hitachi TM1000/ Tabletop Microscope).

3. Results and Discussion

3.1. Gravimetric techniques: Concentration and immersion time effect

Gravimetric techniques evaluated the corrosion rate (CR) and inhibition efficiency (IE%) of the inhibitor at various concentrations (0.001–0.005 M) and immersion periods (5, 10, 24, and 48 h) at 303 K, and the data are demonstrated in Figure 2. The corrosion rates and inhibition efficiency (%) were evaluated according to equations (1) and (2).

$$C_R = \frac{\Delta W}{St} \quad (1)$$

$$IE(\%) = \frac{C_{Ra} - C_{Rp}}{C_{Ra}} \times 100 \quad (2)$$

Where ΔW is the weight loss, S is the exposed area (cm^2), t is the exposure time (h), and C_{Ra} and C_{Rp}, are the corrosion rates without and with DP, respectively.

The corrosion rate decreased, and inhibition efficiency increased with increasing the inhibitor concentration, as demonstrated in Figure 2 [23-26]. Therefore, it is clear that the corrosion rate and inhibition efficiency were concentration-dependent. If the inhibitor concentration increases, more molecules are adsorbed on the metal surface, resulting in improved inhibition effectiveness. The adsorbed inhibitor molecules block the sites of reaction and shield the surface of mild steel from being corroded. As the azomethine (imine) had ample available electrons, lone pairs of nitrogen atoms, and pi-electrons, which are coordinated to the iron atoms of mild steel surface through unoccupied d-orbital, they could prevent and control corrosion. At concentrations of 0.005 M and 303 K, DP showed the highest inhibition efficiency of 94.8% for the immersion time five hours. Beyond this, no improvement in inhibition effectiveness has been found with a further increase in inhibitor concentration. Increasing the immersion time leads to a higher corrosion efficiency. It reduces the corrosion rate, but when the immersion time reaches 24 hours, the corrosion efficiency decreases, and the corrosion rate increases. The possible reason for this is that the metal's surface dissolves due to a lack of inhibitor particles adsorbed on the surface of the metal [27-31].

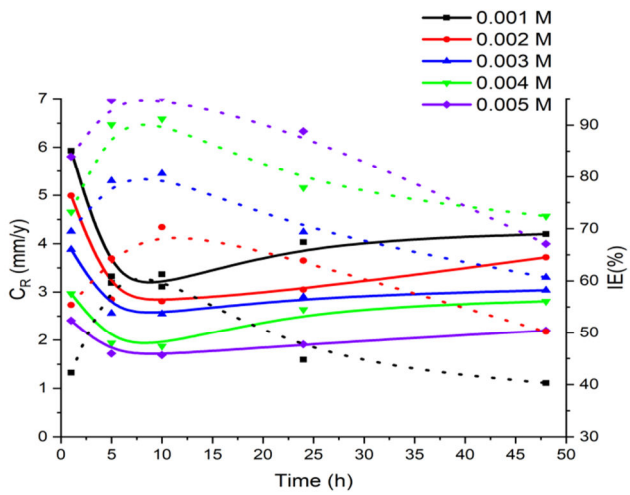

Figure 2: Weight loss data of mild steel after a period of immersion (1, 5, 10, 24, and 48 h) in 1 M HCl in the absence and presence of various inhibitors' concentrations at 303 K.

3.2. Gravimetric techniques: Effect of temperature

Temperature significantly influences the corrosion rate and inhibition efficiency, and with increasing temperature, the rate of corrosion increases in a corrosive environment. To understand the inhibition efficiency at higher temperatures, the weight loss measured from 303-333 K. The inhibition efficiency of the investigated inhibitor gradually decreased with an increase in temperature, as demonstrated in Figure 3. The studied inhibitor demonstrated low inhibition efficacy at higher temperatures because the rising temperature did not support physical interactions, thereby decreasing inhibition efficiency. The process of adsorption and desorption occurs at higher temperatures after a brief period. Finally, the increasing immersion time of mild steel surface in hydrochloric acid solution will decrease the inhibition effectiveness [32, 33].

In order to study the inhibition mechanism, gravimetric techniques were taken at different temperatures without and with the addition of various concentrations of 2-(2,4-dimethoxybenzylidene)-N-phenylhydrazinecarbothioamide (DP) at various exposure time. In the investigated temperature extent (303-333 K), the corrosion rate increases with increasing solution temperature, and the inhibition efficiency of 2-(2,4-dimethoxybenzylidene) N-phenylhydrazinecarbothio-amide is decreased. 2-(2,4-dimethoxy-benzylidene)-N-phenylhydrazinecarbothio-amide acts as an excellent corrosion inhibitor of mild steel in a 1 M hydrochloric acid environment. The highest inhibitive efficiency is 94.8% at 0.005 M for 5 hours as exposure time 303 K. The value of inhibition efficiency increase with the inhibitor concentration but decreases with increasing immersion time and solution temperature of mild steel corrosion 1 M hydrochloric acid environment.

3.3. Adsorption isotherms

The interaction between the inhibitor molecules and the surface of mild steel was investigated by the adsorption isotherms resulting from physical or chemical adsorption. Generally, the corrosion rate is affected by the coverage degree of inhibitor molecules surface. Therefore, the inhibitive performance is described as the electrode surface's function covered by the inhibitor molecules. The surface coverage (θ) and concentration of the tested inhibitor were utilized to evaluate the adsorption isotherm linear relation of the (θ = IE (%) / 100). Different adsorption isotherms, such as Frumkin, Temkin, and Langmuir, were investigated for the best conception of the inhibitor molecules behavior, and the Langmuir model was the best one [34].

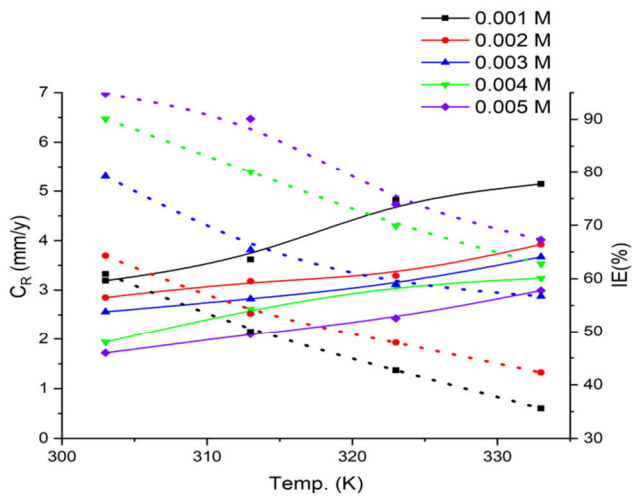

Figure 3: Weight loss data of mild steel after immersion time (5 h) in 1 M HCl in the absence and presence of various inhibitor concentrations at 303 - 333 K.

The relation between surface coverage and concentration was determined according to equation 3:

$$\frac{C}{\theta}=\frac{1}{K}+C \quad (3)$$

Where K is the equilibrium constant.

In the plot between surface coverage and concentration, a straight line with an R-value near one was obtained, as demonstrated in Figure 4. The linear relation suggests that the inhibitor molecules adsorbed on the coupon surface follow the Langmuir isotherm model [35].

K_{ads} was evaluated utilizing equation (3), and the adsorption free energy was determined from K_{ads} according to equation (4).

$$\Delta G^{o}_{ads}=-RT\ln(55.5K_{ads}) \quad (4)$$

Where T is the temperature, R is the universal gas, and the number 55.5 mol.dm^{-3} in the water constant. ΔG^{0}_{ads} determine utilizing equation (4), and it was -39.26 kJ.mol^{-1}. These values suggested that the inhibitor molecules adsorbed physisorption and chemisorption on the mild steel surface. It has been published that if free energy value around -20 kJ.mol^{-1} means physisorption, and free energy value around -40 kJ.mol^{-1} indicates chemisorption [36].

3.4. Inhibition mechanism

The inhibition effect of DP towards mild steel corrosion in a 1 M hydrochloric acid environment may be attributed to the adsorption of DP molecules in the mild steel-environment. The adsorption of DP molecules is affected by the alloy's physical properties, inhibitor structure, corrosive solution type, temperature, and mild steel surface. Generally, the inhibition efficiency value depends on the density of electrons at the inhibitor molecules' active sites. Chemical adsorption of DP molecules arises from the interactions between the unoccupied electrons of heteroatoms and pi-electrons with empty Fe-d-orbital. The inhibition impact is attributed to the interaction of heteroatoms of DP molecules iron atoms on the mild steel surface, which produce coordination bonds between them and forming an insoluble, stable, and uniform thin film. The significant inhibitive DP performance is due to electron-donating one sulfur atom, two oxygen atoms, three nitrogen atoms, and the C=N group and the aromatic rings, which provides a high, and it was found that inhibition efficiency increases with electron density.

3.5. Morphological investigation

To prove the tested inhibitor's adsorption on the surface of the metal in the absence and presence and tested inhibitor at the concentration (0.005) in 1 M hydrochloric acid solution, scanning electron microscopy (SEM) tests were conducted. Figure 5 shows the SEM micrograph of surface coupons at 303 K. Figure (5a) is the coupon surface without the inhibitor. In contrast, Figure (5b) is the micrograph of the coupon surface after five hours of immersion in the corrosive environment. It is evident from the SEM micrograph (Figure (5a)) that the coupon surface was highly corroded because of the HCl solution. It is evident from Figure (5b) that the coupon surface is

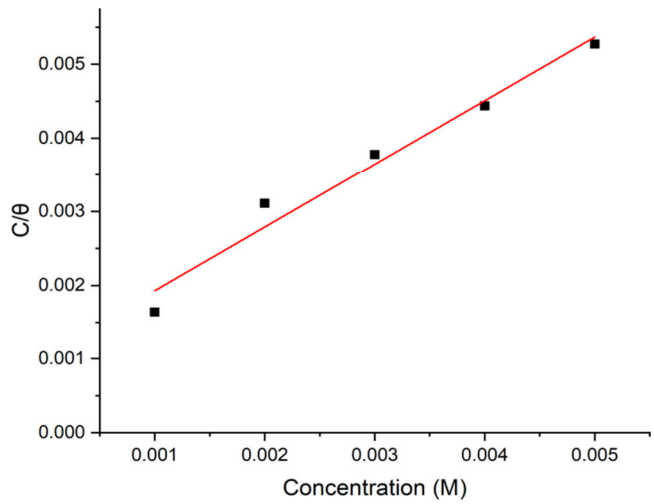

Figure 4: Langmuir isotherm for the adsorption of DP molecules on coupon surface in 1 M HCl at 303 K.

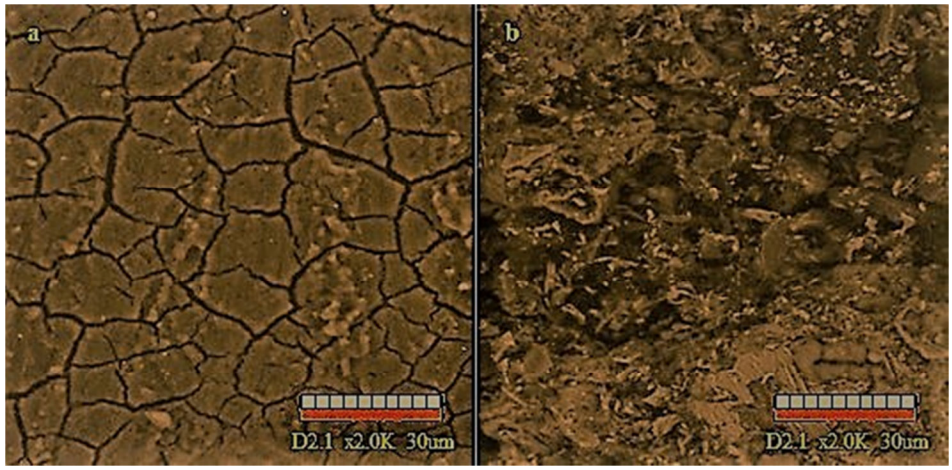

Figure 5: SEM micrograph of coupons surface after five hours immersion in 1 M HCl at 303 K: (a) without and (b) with DP.

smooth with no significant changes, which confirmed that the DP can prevent corrosion of the corrosive environment.

3.6. Comparison with other inhibitors

The present anti-corrosive compound is the product of a reaction of 2,4-dimethoxybenzaldehyde and N-phenylhydrazinecarbothioamide included in the synthesis of some pharmaceuticals. Hence, the resulting inhibitor is an eco-friendly compound. Moreover, the new inhibitor is produced from a one-step reaction of cheap raw materials and can thus be produced industrially. It has been well documented in the literature that organic inhibitors are more effective and inexpensive than inorganic inhibitors. Recently, a lot of research has been devoted to producing non-toxic green inhibitors. In this study, anti-corrosive light steel inhibitors were synthesized and compared with different organic inhibitors concerning their inhibition efficiency, which was evaluated from weight loss techniques, in an HCl medium. As noted from Table 1, the studied inhibitor DP has high inhibition efficiency compared to corrosion inhibitors and the ability to manufacture easily and in one step. Although some of the corrosion inhibitors, composite in Table 1, have relatively higher inhibition efficiencies than the ones examined, they are either toxic, environmentally unfriendly, or expensive to formulate, with reactions that require more than one step and require costly solvents.

Lately, a lot of studies have been devoted to providing eco-friendly inhibitors. In this investigation, an anti-corrosive mild steel inhibitor was synthesized and examined with different organic inhibitors concerning their inhibition efficiency, assessed by weight loss measurements, in an acidic medium. As noted in Table 1, the studied inhibitor has high inhibition efficiency compared to corrosion inhibitors and the ability to manufacture easily and in one step. Although some of the corrosion inhibitors, composite in Table 1, have relatively higher inhibition efficiencies than the one examined, they are either toxic, environmentally unfriendly, or expensive to formulate, with reactions that require more than one step and require costly solvents.

4. Conclusion

A new corrosion inhibitor was synthesized and investigated its ability as a corrosion inhibitor for mild steel in hydrochloric acid solution. The studied inhibitor demonstrated significant inhibitive performance for mild steel in 1 M HCl. The gravimetric techniques showed that the inhibitive efficiency increased with an increase in the synthesized inhibitor concentration and decreased with an increase in temperature. The investigation of adsorption isotherm revealed chemosorption and physisorption, and the isotherm obeyed Langmuir. Moreover, the SEM micrograph confirmed the formation of a film on the metal surface, which supports this inhibitor's corrosion inhibition performance.

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