

Comparative Investigations of the Corrosion Inhibition Efficiency of a 1-phenyl-2-(1-phenylethylidene)hydrazine and its Analog Against Mild Steel Corrosion in Hydrochloric Acid Solution

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ABSTRACT

The corrosion inhibition performance of 1-phenyl-2-(1-phenylethylidene)hydrazine (PPEH) and 1-(1-(4-methoxyphenyl)ethylidene)-2-phenylhydrazine (MPEH) for mild steel (MS) in (1.0 M) hydrochloric acid was investigated based on weight loss measurements. Conditions that affect the corrosion inhibition efficiency, such as concentration, immersion time, the chemical structure of Schiff bases, and solution temperature, were possessed in the current study. The experimental findings demonstrated that PPEH is less than MPEH. In addition, (0.005 M) PPEH protected the tested surface by (83.8%) at (30 °C), while (0.004 M) MPEH afforded (87.2%) protection. The inhibition efficiencies of PPEH and MPEH are (83.8%) and (95.1%), respectively, at a concentration of (0.005 M) at (5 h), exposure time. The efficiency of corrosion inhibition of MPEH is greater than PPEH in the hydrochloric acid environment due to the electron-donating group (methoxy group) substituted to the para-position of the phenyl ring of MPEH. The desorption process of both studied inhibitors upon the (MS) surface follows the Langmuir adsorption isotherm. Scanning electron microscopy (SEM) photographs proved the protective layer's formation upon the (MS) surface. Prog. Color Colorants Coat. 15 (2022), 53-63© Institute for Color Science and Technology.

1. Introduction

Steel corrosion is one of the main economic and safety concerns of many industrial processes, such as oil and gas manufacturers and construction [1, 2]. Generally, mild steel is a broadly utilized material in most industrial processes because of its relatively cheap and significant mechanical characteristics [3, 4]. Corrosion inhibitors are used for protecting the (MS) against corrosion in different environments [5-7]. It is an easy technique of controlling or retarding the rate of corrosion, and it is a

low cost [8, 9]. Organic compounds containing heteroatoms, such as nitrogen, oxygen, sulfur, phosphorous, and molecules with double bonds systems, are usually considered inhibitors for metal corrosion and have significant inhibition activities [8-10]. Schiff bases as-synthesized compounds were manifested as significant corrosion inhibitors with high inhibitive efficiencies at low and high concentrations [11-13]. In recent years, Schiff bases have of great interest because they are considered effective inhibitors for (MS)

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corrosion since they donate considerable inhibition compared to starting materials such as carbonyls and amines [14-18]. An azomethine (imine) is an active site with a nitrogen-carbon double bond and a Schiff base. The imine site existence in the Schiff bases consolidates their capability of adsorption and inhibitive performance against corrosion [19, 20]. It is necessary to complete the synthesis of new organic compounds' as effective corrosion inhibitors in corrosive environments [21-39]. Accordingly, the current study refers to the corrosion inhibition properties of two synthesized Schiff bases, namely 1-phenyl-2-(1-phenylethylidene) hydrazine (PPEH) and 1-(1-(4-methoxyphenyl)ethylidene)-2-phenylhydrazine (MPEH) for (MS) in hydrochloric acid.

For this reason, gravimetric methods and scanning electron microscopy (SEM) analysis were utilized. The effect of different concentrations, immersion time, and temperatures was investigated. Also, the various thermodynamic and kinetic parameters are evaluated. The weight loss and SEM results were correlated to describe the inhibitors' coordination mechanism with a steel surface. This work's novelty lies in the use of new synthesized organic compounds as new corrosion inhibitors for the protection of mild steel in the corrosive environment. The benefit of newly synthesized inhibitors is their high inhibition efficiencies (83.8% and 95.1%), low inhibitor concentrations, and long immersion time.

2. Experimental

2.1. Materials

The chemical composition of the utilized (MS) coupons for conducting corrosion measurements is depicted in Table 1. Hydrochloric acid (37%, Sigma-Aldrich Malaysia) was used to prepare (1 M) hydrochloric acid environment by diluting double distilled water. All the gravimetric measurements were conducted in (1 M) HCl environment comprising (0.001, 0.002, 0.003, 0.004, and 0.005 M) of

synthesized inhibitors (PPEH and MPEH). Before each experimental process, the mild steel surface of tested coupons was subjected to emery papers to polish it, and finally, it was rinsed twice by Distilled water and Acetone.

2.2. Synthesis of inhibitors

The two Schiff bases were synthesized by refluxing phenylhydrazine (0.01 mol) with acetophenone or p-methoxyacetophenone (0.01 mol) into ethyl alcohol (50 mL) using glacial acetic acid as a catalyst for (10 h) (Figure 1). TLC monitored the completion of the reaction. Thus, the solid precipitate obtained was filtered and dried. The inhibitors were re-crystallized using ethyl alcohol. For the first corrosion inhibitor, namely 1-phenyl-2-(1-phenylethylidene) hydrazine (PPEH), the yield was 62%. Molecular formula: $C_{14}H_{14}N_2$. Elemental analysis (Computed): C, 79.97; H, 6.71; N, 13.32. Found: C; 80.06, H; 6.69, N; 13.61%. FTIR (KBr, solid-state; cm^{-1}): $\nu(N-H)$ 3254.5, $\nu(C-H$ aromatic) 3029.7, and $\nu(imine)$ 1604.3. 1H NMR (400 MHz, $CDCl_3$): δ 2.49 ppm (s, 3H (CH_3)), δ 9.72 ppm (s, 1H, (NH amine)), δ 7.51-7.81 ppm (m, 5H (aromatic ring)), δ 6.96-7.34 ppm (m, 5H (aromatic ring)). ^{13}C NMR (400 MHz, $CDCl_3$): δ 16.7, 114.3, 124.2, 128.3, 129.8, 131.4, 138.1, 144.8, and 165.3. For the second corrosion inhibitor namely 1-(1-(4-methoxyphenyl)ethylidene)-2-phenylhydrazine (MPEH), the yield was 57%. Molecular formula: $C_{15}H_{16}N_2O$. Elemental analysis (Calculated): C, 74.97; H, 6.71; N, 11.66. Found: C; 75.23, H; 6.73, N; 12.09%. FTIR (KBr, solid state; cm^{-1}): $\nu(N-H)$ 3249.7, $\nu(C-H$ aromatic) 3028.6 and $\nu(imine)$ 1604.1. 1H NMR (400 MHz, $CDCl_3$): δ 2.51 ppm (s, 3H (CH_3)), 3.44 (s, 3H, methoxy group), δ 9.76 ppm (s, 1H, (NH amine)), δ 7.37-7.75 ppm (m, 5H (aromatic ring)), δ 6.91-7.28 ppm (m, 4H (aromatic ring)). ^{13}C NMR (400 MHz, $CDCl_3$): δ 17.74, 115.1, 124.5, 128.8, 131.4, 131.7, 139.3, 145.2, 161.4, and 165.9.

Table 1: The mild steel chemical composition.

| Carbon | Manganese | Silicon | Aluminum | Sulfur | Phosphorus | Iron |
|--------|-----------|---------|----------|--------|------------|--------|
| 0.21% | 0.05% | 0.38% | 0.01% | 0.05% | 0.09% | 99.21% |

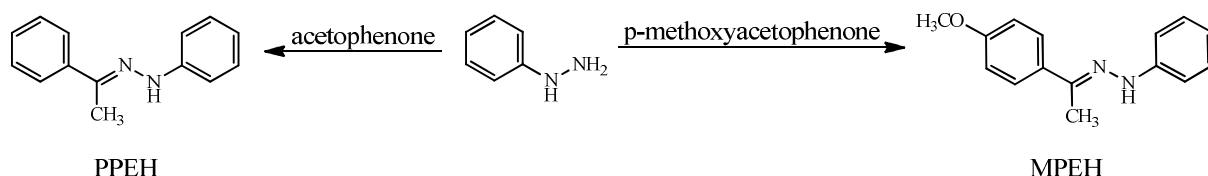


Figure 1: Synthesis scheme of inhibitors.

2.3. Weight loss measurements

Analysis of weight loss was conducted utilizing (MS) coupons of size ($4.5 \times 2 \times 0.025 \text{ cm}^3$). All calculations of weight loss were carried out in (1 M) hydrochloric acid solution as per ASTM G1 [40] with studied inhibitor concentrations of (0.001-0.005 M) at (30 °C). A gravimetric measurement was conducted at 30 °C, which was selected as room temperature. The coupons were immersed into various corrosive environments. After exposure times (1, 5, 10, 24, and 48 h) of measurements, steel coupons were accurately weighed. The experimental processes were conducted by keeping the temperature constant, and a thermostat was utilized for this reason. Gravimetric investigations were conducted at different temperature degrees (30, 40, 50, and 60 °C) to know the corrosion phenomena' temperature effect. The steel coupons weight loss was determined, nearly the weight variation in the studied inhibitors' absence and presence in hydrochloric acid medium. All the experimental processes were triplicate conducted to obtain accurate findings [40]. The rate of corrosion (CR ; mm.y^{-1}) and Inhibition efficiency ($\text{IE}\%$) can be determined from gravimetric analysis according to equations (1) and (2) [28, 35].

$$\text{IE } \% = \frac{w_0 - w_i}{w_0} \times 100 \quad (1)$$

Where w_0 and w_i represent the (MS) coupon weight loss into (1 M) HCl and the (MS) coupon weight loss bearing different inhibitor concentrations, respectively.

$$C_R = \frac{87.6w}{atp} \times 100 \quad (2)$$

Where w represents the (MS) coupon weight loss (mg), a represents the (MS) coupon area (cm^2), t represents the time of immersion (h), and ρ represents the utilized (MS) coupon density (g/cm^3).

2.4. Scanning electron microscopy (SEM)

Inspection of the surface of (MS) coupon in the presence and absence of the optimal concentration of studied inhibitors (0.005 M) inundated for (5 h) at (303 K) was investigated by electron microscopy TM1000 Hitachi Tabletop Microscope model.

3. Results and Discussion

3.1. Measurements of weight loss

Phenylhydrazine and acetophenone (or p-methoxyacetophenone) were refluxed in the existence of an acid catalytic quantity for affording the equivalent Schiff bases, namely 1-phenyl-2-(1-phenylethylidene)hydrazine (PPEH) and 1-(1-(4-methoxyphenyl)ethylidene)-2-phenylhydrazine (MPEH), as evinced in Scheme 1. The spectra of proton nuclear magnetic resonance of PPEH and MPEH revealed the presence of a (1 H) singlet at (9.72 ppm) and (9.76 ppm) due to the azomethine groups, respectively. The spectra of carbon-13 nuclear magnetic resonance of PPEH and MPEH revealed a singlet at (165.3 ppm) and (165.9 ppm), respectively, which were related to $\text{R}_2\text{C}=\text{N}$.

The corrosion rate-time and the inhibition efficiency-time curves of (MS) in the (1 M) hydrochloric acid environment were evaluated in nonexistence and existence of the different inhibitor (PPEH or MPEH) concentrations, which are demonstrated in Figures 2 and 3. The rate of corrosion of the (MS) was determined in nonexistence and existence of the synthesized Schiff base inhibitors at various concentrations (0.001, 0.002, 0.003, 0.004, and 0.005 M) in (1 M) HCl solution as corrosive surrounding at (303 K). The corrosion rates determined in (mmpy) demonstrated that the corrosion rate reduced considerably in the presence of PPEH or MPEH as-synthesized inhibitors. The highest corrosion rate was found in the absence of the inhibitor (77.2 mmpy).

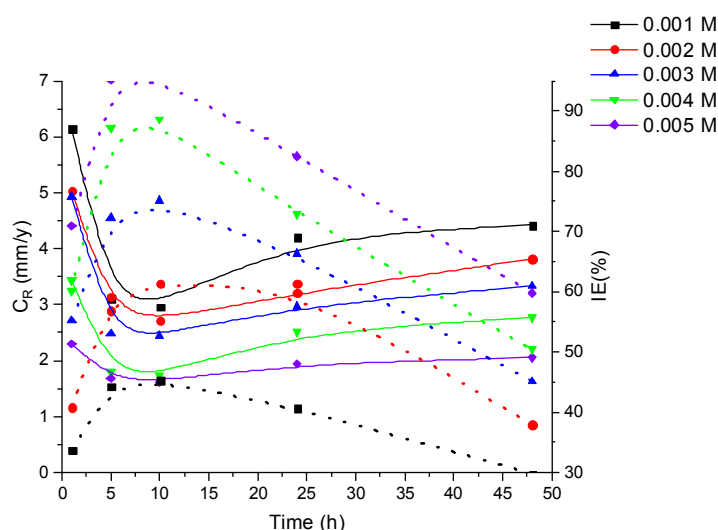


Figure 2: Variation of the rate of corrosion rate and the efficiency of inhibition for (MS) into (1 M).

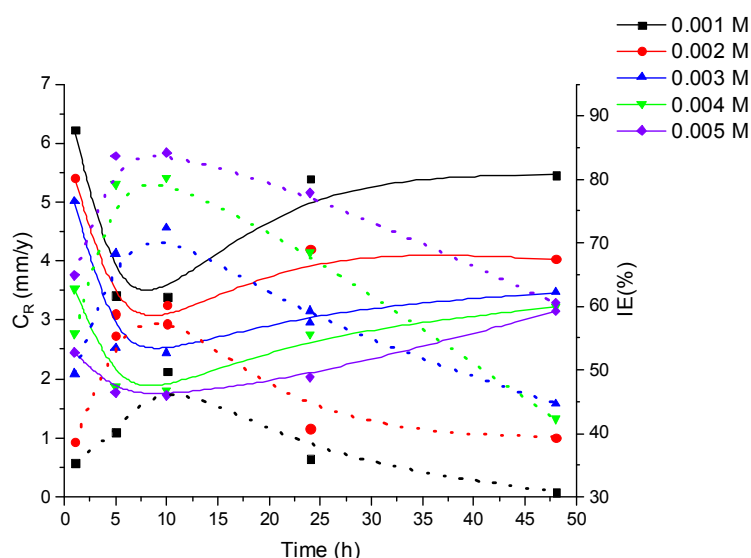


Figure 3: Variation of the rate of corrosion rate and the efficiency of inhibition for (MS) into (1 M) hydrochloric acid solution at 1-48 h immersion, 303 K, with PPEH concentration 0.001-0.005 mM.

It's evident from the findings that the loss of weight declined, and the $\eta\%$ raised with a subsequent increase in the studied inhibitor's concentration. The overall inhibition efficiency of MPEH and PPEH was 95 % and 83 %, respectively, which were achieved at the concentration of 0.005 M. The differences in the rate of corrosion and the inhibition efficiency with different concentrations of (a) MPEH and (b) PPEH into (1 M) hydrochloric acid environment with various times of immersion at 303 K are demonstrated in Figures 2 and 3, respectively.

It is evident from the postulated Figures 2 and 3 that the corrosion rate was the highest with the first inhibitor concentration in an acid environment but significantly

decreased with the increase of inhibitor concentration. Without an inhibitor, the mild steel coupon corroded at a rate of 77.2 mm/y but was reduced to 1.6 mm/y and 1.8 mm/y upon the addition of 0.005 mM MPEH and PPEH, respectively. This translated to the inhibition efficiencies of 95 and 83 % by 0.005 mM MPEH and PPEH, respectively. This observation might be attributed to the blocked off the corrosion reaction sites by adsorbed corrosion inhibitor molecules, which impedes or controls the corrosion process [41]. However, it is observed that the influence of PPEH and MPEH is a function of concentration and time. The rate of corrosion reduces, while the efficiency of inhibition increases with a rise in the PPEH and MPEH concentration at

immersion time below (24 h). This might be credited the higher coverage of the surface of (MS) [42].

A close observation of Figures 2 and 3 elucidates that the corrosion rate and the inhibition efficiency remained almost constant when the immersion time was increased from 5 h to 10 h. For instance, the inhibition efficiencies at the immersion times 5 h and 10 h for MPEH are more than 95 %, whereas, for PPEH, they are around 87 %. It suggests that the inhibitor layer covered the mild steel surface when the immersion time was 5 h, and further immersion time had a minimal effect on the inhibition performance. Moreover, at a long immersion time, the inhibition efficiency decreased due to the dissolution of the inhibitor layer, which covered the surface of (MS).

3.2. Effect of temperature

The corrosion process parameters were obtained to assess the stability of adsorbed inhibitor molecules layer on (MS) surface. Besides, gravimetric techniques were performed over the temperature range of 303-333 K without and with MPEH (or PPEH) at the studied concentrations 0.001-0.005 mM and the optimum

immersion time (5 h). The findings obtained are demonstrated in Figures 4 and 5. These figures indicate that the efficiency of inhibition reduces by raising the temperatures for the two tested inhibitors (MPEH and PPEH). This is due to the elevated corrosion rate and the incomplete desorption of inhibitor molecules from the (MS) surface with the temperature.

Additionally, inhibitive MPEH and PPEH were investigated at different temperatures (303, 313, 323, and 333 K). MPEH shows effective inhibition with a high inhibition performance: 95.1%, 89.3%, 77.2%, and 73.8%, as depicted in Figure 4. PPEH inhibition efficiencies are 83.7%, 80.7%, 72.4%, and 68.5%, as revealed in Figure 5. Figures 4 and 5 display that if the temperature rises, then the efficiencies of the inhibition of MPEH and PPEH decrease. This can be attributed to physisorption and chemisorption adsorption of MPEH or PPEH molecules on the surface of (MS). In this scenario, the association between the MPEH (or PPEH) molecules and the mild steel surface will form coordination bonds by unshared pairs of electrons of nitrogen atoms and unoccupied d-orbital of the iron atom.

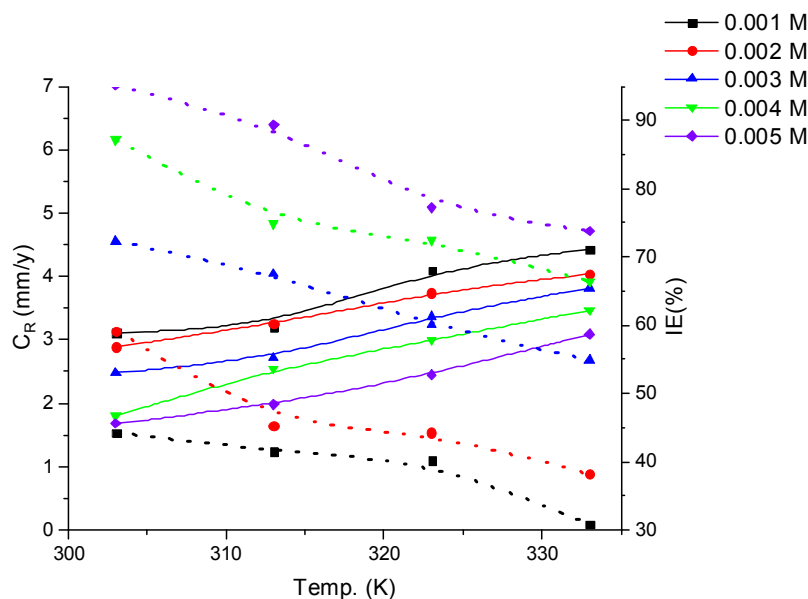


Figure 4: Variation of the corrosion rate and the efficiency of (MS) inhibition into (1 M) hydrochloric acid solution at temperature period 303-333 K, and five hours immersion time with MPEH concentration 0.001-0.005 mM.

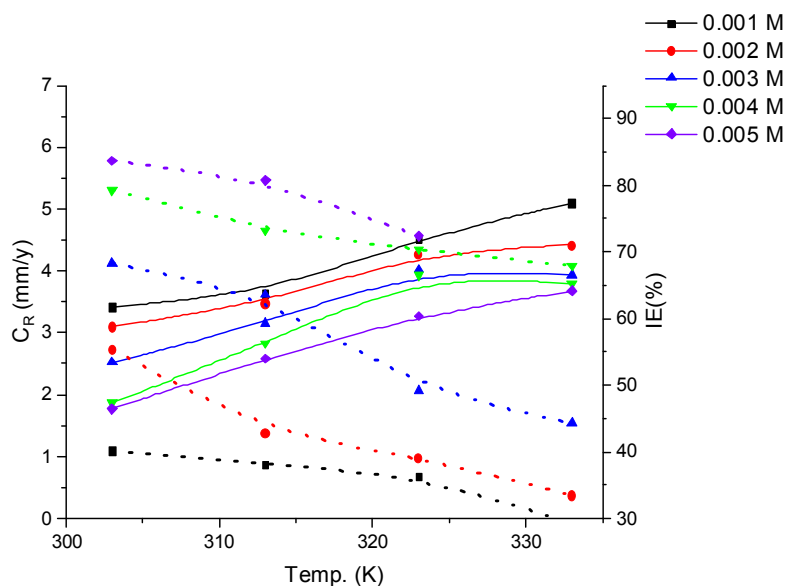


Figure 5: Variation of the corrosion rate and the efficiency of (MS) inhibition into (1 M) hydrochloric acid solution at temperature period 303-333 K, and five hours immersion time with PPEH concentration 0.001-0.005 mM.

3.3. Adsorption isotherms

They can produce significant data about the interactions between the inhibitor molecules and the surface of (MS) [43]. Based on this procedure, different adsorption isotherm models have been recommended [44, 45]. Also, in the present investigation, it is supposed to employ the Langmuir adsorption (equation 4) and Temkin adsorption (equation 5) isotherms to examine the mechanism of adsorption of the tested inhibitors [46-49].

$$\frac{C}{\theta} = \frac{1}{K} + C \quad (4)$$

$$\ln K.C = a.\theta \quad (5)$$

Where:

K: The constant of equilibrium

C: The concentration

θ : The surface coverage

a : The term of lateral interaction

R^2 represents the correlation coefficient, which is utilized to estimate if the adsorption model is harmonious with the methodological findings [50]. It is evident from Figures 6 and 7 that the R^2 values of the plots of \ln concentration vs. surface coverage are significantly different from the unit. They manifest that the MPEH (or PPEH) molecules adsorption upon the

surface of (MS) does not obey the Temkin isotherm. The MPEH (or PPEH) molecules adsorption upon the surface of (MS) follows the Langmuir adsorption. The straight lines between (C) and (C/ θ) are obtained with the correlation coefficient near to one (Figures 6 as well as 7), and the values of slope in the Langmuir formula being nearly equal one. Such outputs verify that the MPEH (or PPEH) values upon the surface follow the Langmuir isotherm. Each molecule of MPEH or PPEH merely is accounting for the single position of adsorption [51-55].

3.4. Morphological surface analysis

The surface investigation was conducted utilizing the SEM techniques for the tested coupons exposed to (1 M) HCl medium in the nonexistence and existence of (0.005 M) of MPEH as-synthesized corrosion inhibitor. The SEM micrographs of the metal surface, (1 M) HCl, and in nonexistence and existence of MPEH are displayed in Figures (8a) and (8b). Considerable damage, as in Figure 8a, was observed on the surface of tested coupons, which was exposed to a corrosive environment indicating that the coupon surface is corroded. Figure (8b) shows that the coupon surface was smooth and not corroded due to the presence of the MPEH at the optimum concentration.

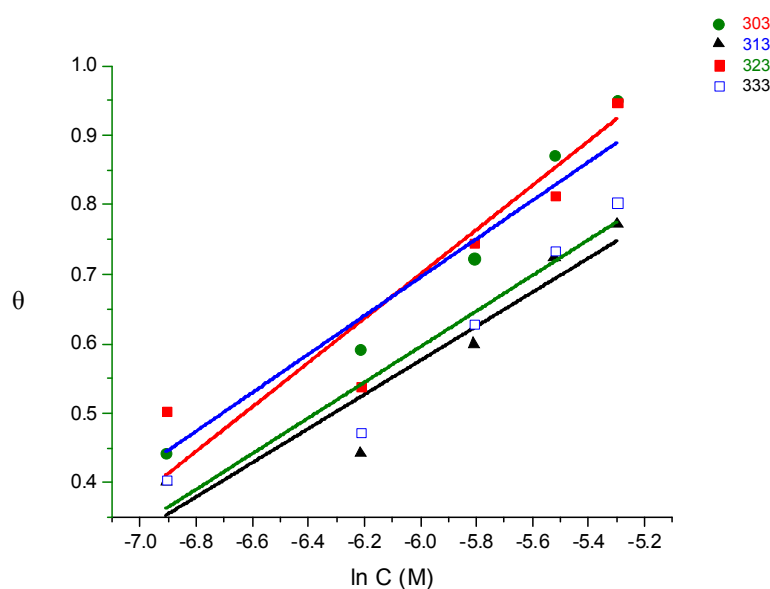


Figure 6: Langmuir's adsorption isotherm of MPEH upon (MS) 's surface at various temperature solutions.

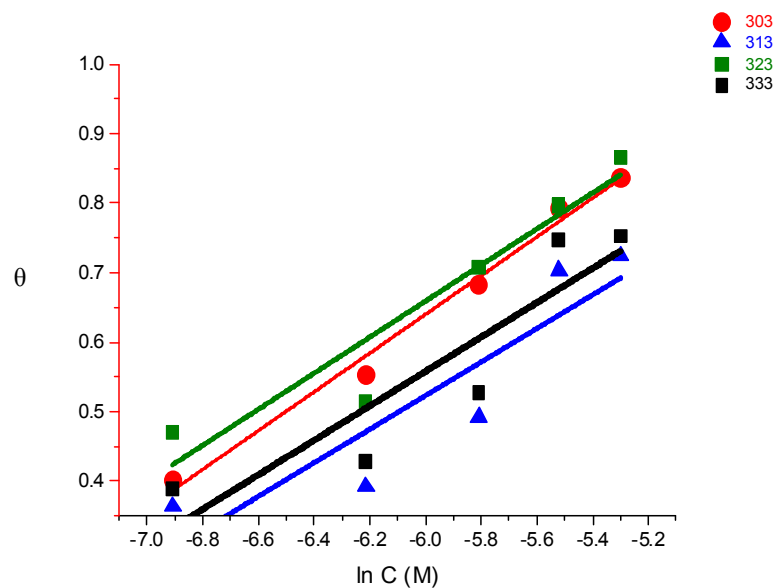


Figure 7: Langmuir's adsorption isotherm of PPEH upon (MS) 's surface at various temperature solutions.

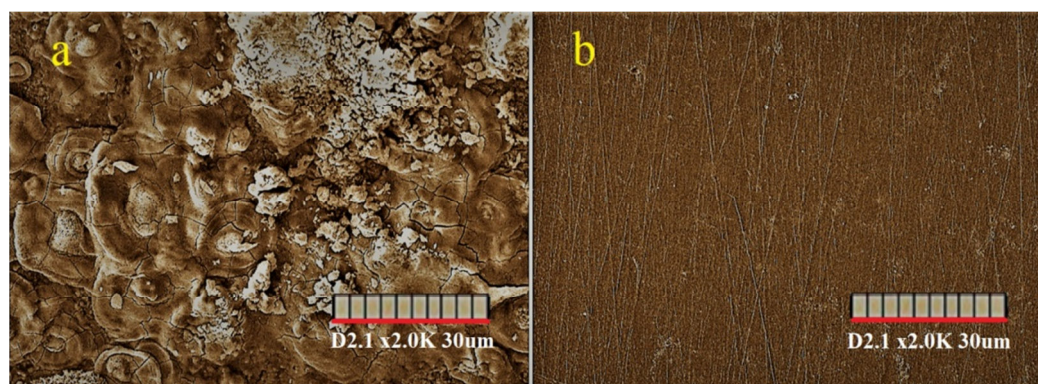


Figure 8: SEM micrographs of (a) mild steel in the absence of MPEH and (b) mild steel in the presence of MPEH.

3.5. Mechanism of inhibition

The mechanism of inhibition is a separation approach, including (i) the inhibitor molecules form a protective layer due to being adsorbed upon the coupon surface and (ii) the molecules of inhibitor form a precipitate upon the coupon surface, acting on the acidic solution to form a protective precipitate [56, 57]. The adsorption process, on the other significant notes, could be generally identified according to the two types of interactions, which are the physical and chemical adsorption [58, 59]:

Physical adsorption includes the electrostatic forces between the ions or the unshared pairs of electrons on the absorbent species and the electrical charge at the mild steel surface/environment interface. The absorption temperature is low, so this absorption is only stable at a relatively low temperature.

Chemical adsorption includes the transfer or the share of charges from an inhibitor to a metallic surface for forming coordination bonds. This adsorption kind is recognized to have extreme absorption energy compared to another adsorption mode. Hence, the coordination bonds are further steady at the higher temperature [60- 64].

4. Conclusion

From the experimental findings, the followings can be concluded:

1) The value of the inhibition efficiency rises with the inhibitor concentration but reduces with an increment in the temperature proposing the physical adsorption.

2) Experimental findings showed that the adsorption process proceeded at the low and high temperatures proposing the chemical adsorption.

3) Langmuir's isotherm gives a helpful characterization of the synthesized corrosion inhibitor's adsorption conduct upon the surface of (MS).

4) Surface morphology investigations revealed that the corrosion of surface metal into (1 M) HCl decreased in the synthesized inhibitor's existence, which resulted in an uncorroded smooth surface.

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