Research Article

Inhibition Effect of Miconazole Nitrate on the Corrosion of Mild Steel in Hydrochloric Acid Medium

J. Ishwara Bhat[1] and Vijaya D. P. Alva[2]

[1] Department of Chemistry, Mangalore University, Mangalagangotri, Karnataka 574199, India
[2] Department of Chemistry, Shree Devi Institute of Technology, Karnataka 575003, India

Correspondence should be addressed to J. Ishwara Bhat, bhatij@yahoo.com

The corrosion inhibition of mild steel by miconazole nitrate, an antifungal drug has been investigated using potentiodynamic polarization, electrochemical impedance spectroscopy technique, and weight loss methods. The experimental results suggested miconazole nitrate is a good corrosion inhibitor for mild steel in 1 M hydrochloric acid medium. The inhibition efficiency increased with increase in inhibitor concentration. The thermodynamic parameters were determined and discussed. The inhibition was assumed to occur via adsorption of the inhibitor molecule on the surface of mild steel following Langmuir adsorption isotherm.

1. Introduction

The corrosion of metals has received prime attention of material scientists and chemists since it has many serious adverse effects including economic, health, safety, technological, and cultural consequences. Corrosion of mild steel is one of the major areas of concern in many industries, where acids are widely used for applications such as acid pickling, acid descaling, and oil well acidizing. Due to general aggressiveness of acid solutions, respective materials of constructions are getting corroded easily. Development of methods to control such corrosion is a challenge to chemists and scientists, working in this area. A large number of methods have been employed to understand the practical problem and its control.

Inhibition is one of the most important applications in the corrosion protection. The use of specific chemical compound as an inhibitor is the best, simple, and practical method to minimize corrosion of metals. The corrosion inhibitors bring down the rate of corrosion to a greater extent even when added in small quantities to the corrosive environment. Inhibitors protect the metals by getting effectively adsorbed on its surface, blocking the active sites of metal dissolution and/or hydrogen evolution, there by retarding overall metal corrosion in aggressive environment.

Organic molecules could be adsorbed on the metal surface by one of the following four mechanisms: (a) electrostatic interaction between charged surface of the metal and the charge of the inhibitor, (b) interaction of unshared electron pairs in the inhibitor molecule with the metal, (c) interaction of π electron with the metal, and (d) a combination of the (a–c) types. Selecting an appropriate inhibitor for a specific environment and the corroding metal is of great importance, since an inhibitor that protects one particular metal may accelerate the corrosion of other. Many heterocyclic compounds containing hetero atoms like N, O, and S have been proved to be effective inhibitors for corrosion of metals in acidic media [1–11]. An assortment of organic compounds having two or more hetero atoms such as N, O, S and multiple bonds in their molecular structure are of particular interest because of their better inhibition efficiency as compared to those containing N or S alone [12]. Most of the inhibitors are toxic and environmentally hazardous. Due to this, interest is still growing to exploit environmentally acceptable corrosion inhibitors. Use of few antibacterial and antifungal drugs has been reported by some investigators [13–16]. Miconazole nitrate (Figure 1) is an azole antifungal drug [17], used in topical antifungal medications for athlete's foot, ringworm, jock itch, and perioral candidiasis [18–20]. Miconazolenitrate is

the commercial name of [(1-(2-(2,4-dichlorobenzyloxy)-2-(2,4-dichlorophenyl)ethyl)-1*H*-imidazole. nitrate]. Perusals of several literatures reveal that there is no information regarding the use of miconazole nitrate as corrosion inhibitor for mild steel. The aim of the present investigation is to study the effect of miconazole nitrate as a potential inhibitor for mild steel in 1 M HCl.

2. Experimental

IS-2062 mild steel samples of Tata steel provided by ABCO Steel International Ltd. Mangalore were used in the present study. The chemical composition of IS-2062 is (wt%): C (0.18%), Mn (0.6%), S (0.05%), P (0.04%), Si (0.1%), and Fe (balance). Test materials were abraded with different emery papers up to 1000 grade, cleaned with acetone, washed with double distilled water, and properly dried prior to exposure. Analar grade HCl and double distilled water were used to prepare all solutions. Miconazole nitrate (F.W. 479.139) from Frapps Chemical Reagent Co. Ltd.) was used for the study.

Weight loss measurements were performed on mild steel coupons with dimensions $1 \times 0.3 \times 0.06$ inches, immersed in 100 mL flask, containing 50 mL of 1 M hydrochloric acid solution with different concentrations of the inhibitor. There was neither agitation nor exposure to oxygen during weight loss measurement. Weight loss of the metal coupons were noted after an immersion period of 24 hours at temperatures ranging from 30°C to 60°C.

Electrochemical tests were carried out with a CH-analyzer model, CH1660D. The cell arrangement used was a conventional three-electrode cell with platinum counter electrode, saturated calomel electrode as reference electrode, and test material (mild steel) as working electrode. The test material was covered by epoxy adhesive-araldite so that only 1 cm^2 area was in contact with the solution. Polarization curves were recorded potentiodynamically, at the scan rate of 1 mV/s, in the range of +250 mV to −250 mV versus OCP potential. Impedance was measured over a frequency range of 1 MHz to 0.05 Hz using amplitude of 10 mV peak to peak using AC signal.

3. Results and Discussion

3.1. Polarization Measurements. Figure 2 shows the potentiodynamic polarization curves for mild steel in 1 M HCl containing 0, 10, 50, 100, and 200 ppm of the miconazole nitrate at 30°C. Corrosion current (i_{corr}) was found to decrease with increase in inhibitor concentration indicating the increased inhibition efficiency with the increase in the concentration of the inhibitor. In acidic solution the anodic process of corrosion is the passage of metal ions from the solid metal to the solution, and the principal cathodic process is the discharge of hydrogen gas or reduction of oxygen. The inhibitor may affect either of them or both anodic and cathodic process.

The decrease in i_{corr} with increase in inhibitor concentration was associated with shift in the corrosion potential (E_{corr}) to more positive value, indicating suppression of
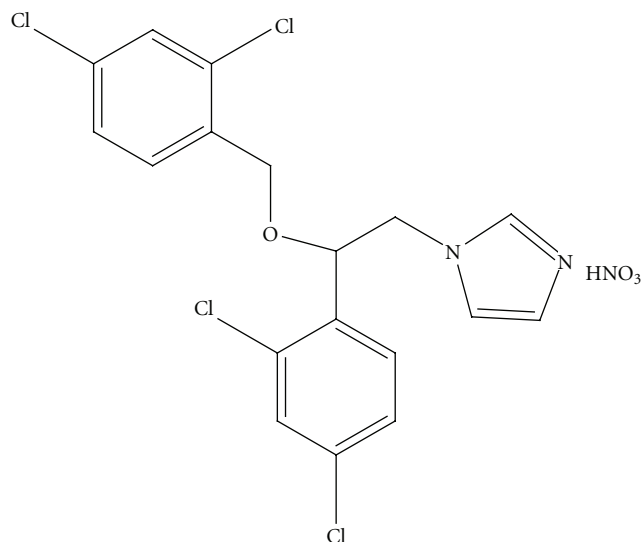

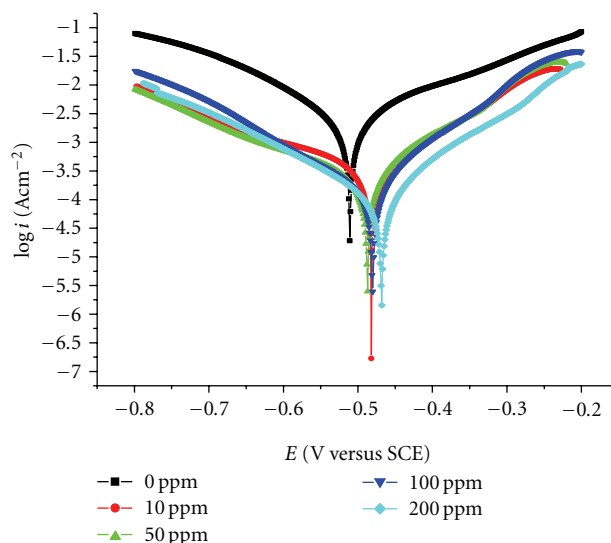FIGURE 1: Molecular structure of miconazole nitrate inhibitor.

FIGURE 2: Potentiodynamic polarization curves of mild steel in 1 M HCl solution at 30°C containing various concentrations of inhibitor.

anodic reaction. Thus inhibitor acts as anodic inhibitor for mild steel in hydrochloric acid medium. The inhibitor gets adsorbed on the steel surface and the adsorbed inhibitor prevents the metal atoms from participating in the anodic reaction of corrosion. The values associated with electrochemical parameter and calculated inhibition efficiency (I.E%) for mild steel is given in Table 1. The inhibition efficiency is given by [21]

$$\text{I.E(\%)} = \left(1 - \frac{i'_{corr}}{i_{corr}} \right) \times 100, \qquad (1)$$

where, i'_{corr} and i_{corr} are the corrosion currents in the presence and absence of the inhibitor, respectively.

TABLE 1: Electrochemical parameters of mild steel in 1 M HCl solution at 30°C.

(inhibitor)/ppm	$i_{corr} \times 10^{-4}$ (A)	$C_R \times 10^2$	E_{corr} (V)	βa (V/dec)	βc (V/dec)	I.E (%)
0	28.1	13.2	−0.5105	5.106	7.357	—
10	4.992	2.279	−0.4816	7.108	3.772	82.23
50	3.833	1.75	−0.4872	7.124	4.237	86.35
100	2.048	0.9349	−0.4804	8.751	6.894	92.71
200	1.273	0.5813	−0.4679	8.717	6.996	95.46

TABLE 2: AC impedance data of mild steel in 1 M HCl solution at 30°C.

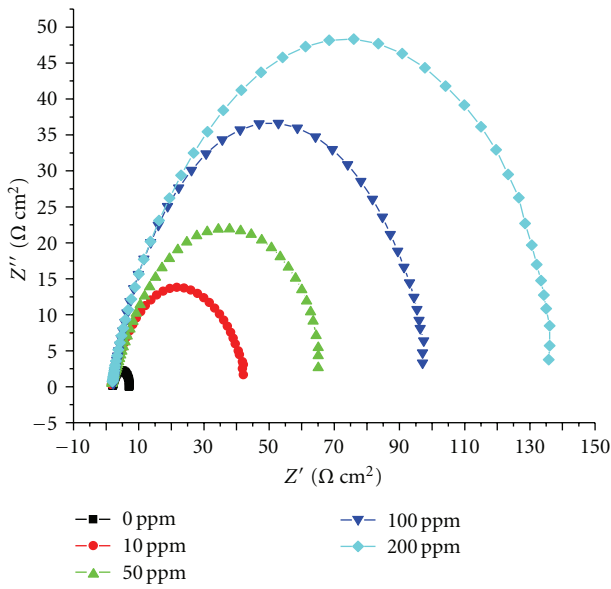| (inhibitor)/ppm	R_p	I.E (%)	C_{dl}
0	7.17	—	318.24
10	41.77	82.83	255.55
50	66	89.13	205.05
100	96.75	92.58	175.74
200	135.6	94.71	169.61

FIGURE 3: Nyquist plots for mild steel in 1 M HCl solution at 30°C containing various concentrations of inhibitor.

Corrosion rate (C_R) in mpy (mils penetration per year) is calculated using [22]

$$C_R = K \frac{ai}{nD}, \tag{2}$$

where a is the atomic weight of the metal, i is the current density in A/cm^2, n is the number of electrons lost, D is the density in g/cm^3, and K is a corrosion constant depending on the unit of corrosion rate (for mpy, $K = 1.288 \times 10^5$).

3.2. Electrochemical Impedance Spectroscopic Measurements (EIS). The impedance spectra (Nyquist plots) of mild steel in 1 M HCl containing 0, 10, 50, 100, and 200 ppm of the

miconazole nitrate at 30°C is shown in Figure 3 Nyquist plots contain depressed semicircle with centre under real axis. The size of the semicircle increases with the inhibitor concentration, indicating the charge transfer process as the main controlling factor of the corrosion of mild steel. It is apparent from the plot that the impedance of the inhibited solution has increased with the increase in the concentration of the inhibitor. The experimental result of EIS measurements for the corrosion of mild steel in 1 M HCl in the absence and presence of inhibitor is given in Table 2. As it can be observed from the table, the polarization resistance (R_p) value increased with increase in the concentration of the inhibitor whereas values of the capacitance of the interface (C_{dl}) starts decreasing, with increase in inhibitor concentration, which is most probably due to the decrease in local dielectric constant and/or increase in thickness of the electrical double layer. This suggests that the inhibitor acts via adsorption at the metal/solution interface [23] and the decrease in the C_{dl} values is caused by the gradual replacement of water molecules by the adsorption of the inhibitor molecules on the electrode surface, which decreases the extent of metal dissolution [24].

The inhibition efficiency is given by [25]

$$\%\text{I.E}\left(R_p\right) = \frac{R_{p(inhi)} - R_p}{R_{p(inhi)}} \times 100, \tag{3}$$

where R_p is polarization resistance without inhibitor, and $R_{p(inhi)}$ is polarization resistance with inhibitor.

C_{dl} value is obtained from (4):

$$\omega_{max} = 2\pi f_{max} = \frac{1}{R_p C_{dl}}, \tag{4}$$

where, f_{max} is the frequency at the top of the semicircle (where-Z'' is maximum).

TABLE 3: Corrosion parameters for mild steel in 1 M HCl in the absence and presence of different concentrations of the inhibitor at 30°C by weight loss measurements.

(inhibitor)/ppm	Weight loss (mg cm^{-2})	I.E (%)
0	127.52	—
5	36.86	71.09
10	20.66	83.8
50	14.54	88.6
100	7.22	94.34
200	3.67	97.12
500	1.4	98.9

3.3. Weight Loss Measurements

3.3.1. Effect of Inhibitor Concentration. The weight loss results of mild steel in 1 M HCl in the absence and presence of various concentrations of the inhibitor are summarized in Table 3. The inhibition efficiency was calculated using the following equation:

$$\text{I.E}(\%) = \frac{W_1 - W_2}{W_1} \times 100, \qquad (5)$$

where W_1 and W_2 are weight loss of mild steel in the absence and presence of the inhibitor, respectively.

The surface coverage (θ) was calculated from the following equation:

$$\theta = \frac{W_2 - W_1}{W_2}. \qquad (6)$$

The corrosion rate (C_R) in mils penetration per year (mpy) is calculated using the formula [22]

$$C_R = \frac{534\ W}{DAt}, \qquad (7)$$

where W is weight loss in milligrams, D is density in grams per cubic centimeter, A is area in square inches, and t is exposure time in hours.

The inhibition efficiency increased with increase in the concentration of the inhibitor for mild steel in 1 M HCl. Results obtained from polarization, EIS and weight loss measurements are in good agreement with each other.

3.3.2. Effect of Temperature. To elucidate the mechanism of inhibition and to determine the thermodynamic parameters of the corrosion process weight loss measurements were performed at 30, 40, 50, and 60°C. The effect of temperature on the corrosion inhibition of mild steel in the presence of the inhibitor is graphically represented in Figure 4.

The slight increase in the inhibition efficiency for corrosion of mild steel at higher temperature can be attributed to the occurrence of chemical adsorption of the inhibitor molecules on its surface. Reported drug, miconazole nitrate has better inhibition efficiency than the already reported antifungal drugs [14–16].
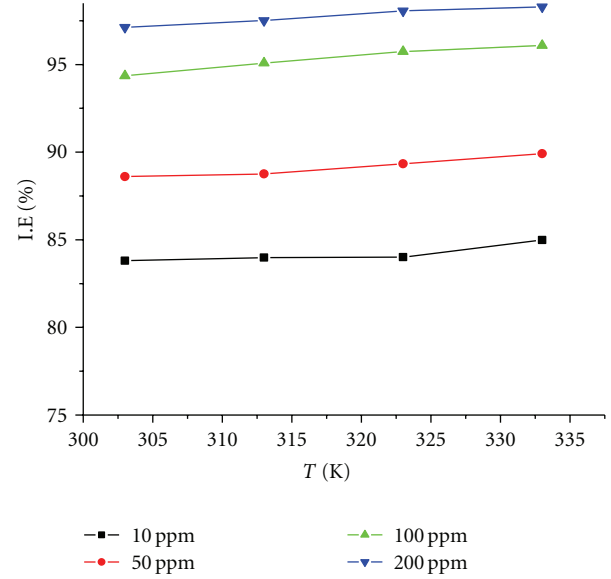

FIGURE 4: Effect of temperature on I.E (%) for mild steel in 1 M HCl in the presence of different concentrations of the inhibitor.
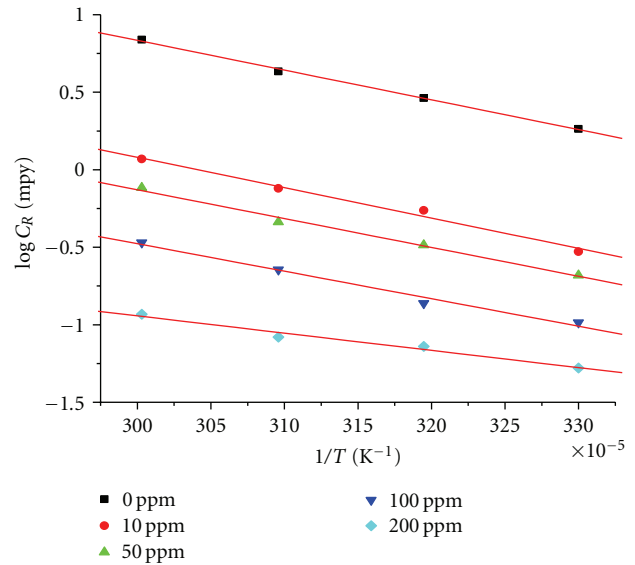

FIGURE 5: Arrhenius plot for mild steel in 1 M HCl solution in the absence and presence of different concentrations of the inhibitor.

3.3.3. Thermodynamic Parameters. The apparent activation energy (E_a°) of metal corrosion in acid media can be calculated from the Arrhenius equation [26]

$$\ln C_R = -\frac{E_a^\circ}{RT} + \text{A}, \qquad (8)$$

where E_a° is the apparent activation energy for the corrosion of mild steel, R is the universal gas constant, A is Arrhenius pre-exponential factor, and T is the absolute temperature. The values of E_a° obtained from the slope of the $\log C_R$ versus $1/T$ plot (Figure 5) are given in Table 4.

TABLE 4: Values of activation parameters for mild steel in 1 M HCl in the absence and presence of different concentrations of the inhibitor.

(inhibitor)/ppm	E_a° (kJ mol^{-1})	ΔH_a° (kJ mol^{-1})	ΔS_a° (J mol^{-1})
0	36.72	34.08	−127.57
10	34.86	32.22	−148.76
50	33.25	30.61	−156.95
100	26.21	23.57	−186.19
200	21.29	18.65	−207.88

The relationship between the temperature dependence of inhibition efficiency of an inhibitor and the activation energy found in its presence was given as follows [27].

(i) Inhibitors whose inhibition efficiency decreases with temperature increase. The value of activation energy (E_a°) found is greater than that in the uninhibited solution.

(ii) Inhibitors whose inhibition efficiency does not change with temperature variation. The value of activation energy (E_a°) does not change with the presence or absence of inhibitors.

(iii) Inhibitors whose inhibition efficiency increases with temperature increase. The value of activation energy (E_a°) found is less than that in the uninhibited solution.

The higher value of activation energy (E_a°) in the presence of inhibitor than in its absence is attributed to its physical adsorption, while its chemisorptions is [28] pronounced in the opposite case [11].

In the present study the lower value of E_a° for mild steel in an inhibitor's presence, compared to that in its absence indicates chemical adsorption. Riggs and Hurd [29] explained that the decrease in activation energy of corrosion at higher levels of inhibition arises from a shift of the net corrosion reaction from that on the uncovered part on the metal surface to the covered one. An alternative form of Arrhenius equation is the transition state equation [26]:

$$C_R = \frac{RT}{Nh} \exp\left(\frac{\Delta S_a^\circ}{R}\right) \exp\left(-\frac{\Delta H_a^\circ}{RT}\right), \quad (9)$$

where, h is Plank's constant, N is Avogadro's number, ΔS_a° is the entropy of activation, and ΔH_a° is the enthalpy of activation. A plot of $\log(C_R/T)$ versus $1/T$ gave a straight line (Figure 6) with a slope of $(-\Delta H_a^\circ/2.303R)$ and an intercept of $[\log(R/Nh) + (\Delta S_a^\circ/R)]$, from which the values of ΔH_a° and ΔS_a° were calculated and listed in Table 4.

The positive values of ΔH_a° for corrosion of mild steel in the presence and absence of the inhibitor reflect the endothermic nature of metal dissolution process. The decrease in ΔH_a° with increase in inhibitor concentration for mild steel corrosion reflected that decrease in mild steel corrosion rate is chiefly decided by the pre-exponential factor [30].

The negative increment in ΔS_a° with increase in the concentration of the inhibitor reveals that decrease in randomness takes place on going from reactant to the activated
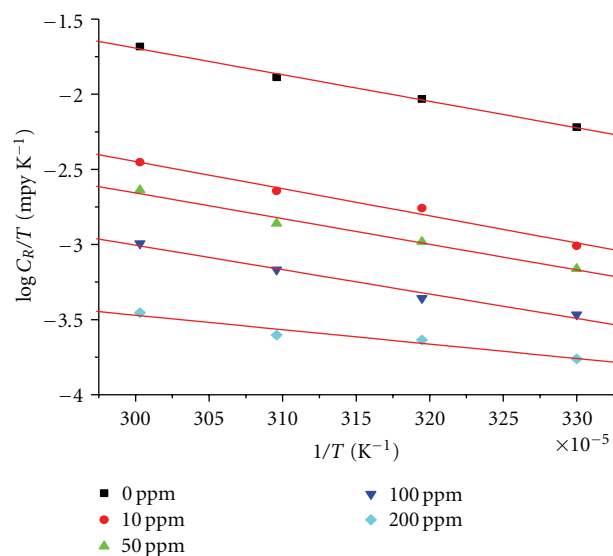

FIGURE 6: Transition plot for mild steel in 1 M HCl solution in the absence and presence of different concentrations of the inhibitor.

complex [31]. This reflects the formation of an ordered stable film of inhibitor on mild steel surface [32].

3.3.4. Adsorption Isotherms. In order to understand the mechanism of corrosion inhibition, the adsorption behavior of the organic adsorbate on the metal surface has to be known. The degree of surface coverage (θ) for different concentration of the inhibitor (c_{inhi}) has been evaluated. The data were tested graphically by fitting to various isotherms. A straight line with correlation coefficient nearly equal to 1.0 was obtained on plotting c_{inhi}/θ against c_{inhi} suggesting adsorption of the compound on the mild steel surface followed Langmuir adsorption isotherm model (Figure 7) at all studied temperatures. According to this isotherm, the surface coverage (θ) is related to inhibitor concentration c_{inhi} [33] by

$$\frac{C_{inhi}}{\theta} = \frac{1}{K_{ads}} + c_{inhi}, \quad (10)$$

where K_{ads} is equilibrium constant of the equilibrium adsorption process. This isotherm assumes that adsorbed molecules occupy only one site and it does not interact with other adsorbed species.

TABLE 5: Values of thermodynamic parameters for the adsorption of the inhibitor in 1 M HCl on the mild steel at different temperatures.

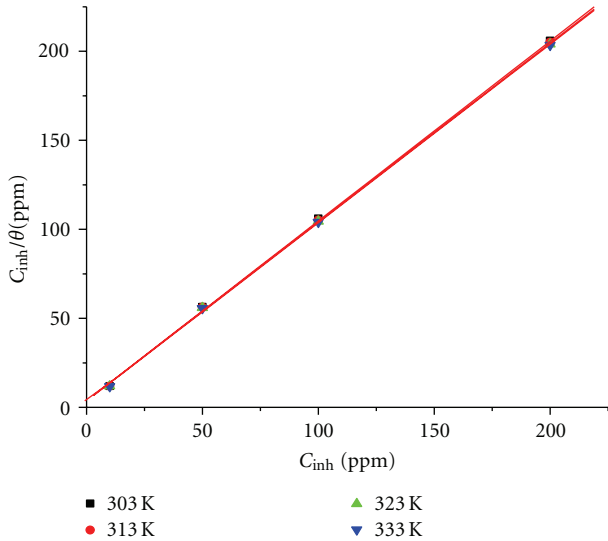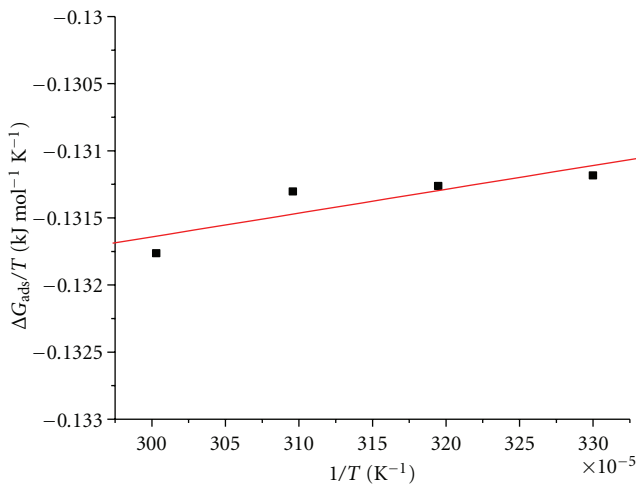| Temperature (K)	$K_{ads} \times 10^4$ (mol^{-1})	ΔG°_{ads} (kJ mol^{-1})	ΔH°_{ads} (kJ mol^{-1})	ΔS°_{ads} (J mol^{-1})
303	12.832	−39.7487		
313	12.954	−41.0852	1.77	136.96
323	13.019	−42.4113		
333	13.758	−43.8771		

FIGURE 7: Langmuir adsorption isotherm for mild steel in 1 M HCl solution at 30°C.

FIGURE 8: $\Delta G^{\circ}_{ads}/T$ versus $1/T$ plot for mild steel in 1 M HCl solution.

The K_{ads} values can be calculated from the intercept lines on the c_{inhi}/θ axis. This is related to the standard free energy of adsorption (ΔG°_{ads}) by the following equation:

$$\Delta G^{\circ}_{ads} = -RT \ln(55.5 K_{ads}), \quad (11)$$

where R is the gas constant and T is the absolute temperature. The constant value of 55.5 is the concentration of water in

solution in mol/L. The calculated values of K_{ads} and ΔG°_{ads} are given in Table 5.

Generally, the magnitude of ΔG°_{ads} around -20 kJ/mol or less negative indicates electrostatic interactions between inhibitor and the charged metal surface (i.e., physisorption). Those around -40 kJ/mol or more negative are indicative of charge sharing or transferring from organic species to the metal surface to form a coordinate type of metal bond (i.e., chemisorptions). In the present work, the calculated values of ΔG°_{ads} at 30°C for mild steel is -39 kJ/mol, which indicated that adsorption of the inhibitor on the metal surface involves complex interactions: both physical and chemical process [34–36]. The negative value of ΔG°_{ads} indicated spontaneous adsorption of the inhibitor on the metal surface. The slight increase in K_{ads} for mild steel showed increased adsorption with increase in temperature.

The enthalpy of adsorption can be calculated from the rearranged Gibbs-Helmholtz equation:

$$\frac{\Delta G^{\circ}_{ads}}{T} = \frac{\Delta H^{\circ}_{ads}}{T} + K. \quad (12)$$

Figure 8 shows the variation of $\Delta G^{\circ}_{ads}/T$ with $1/T$. The plot gave a straight line with a typographical error of $\pm1.65\%$. The slope of straight line, ΔH°_{ads} is given in Table 5. The positive sign of ΔH°_{ads} indicated the endothermic process of adsorption of inhibitor on mild steel. The entropy of adsorption ΔS°_{ads} (Table 5) was calculated using the following thermodynamic equation:

$$\Delta S^{\circ}_{ads} = \frac{\Delta H^{\circ}_{ads} - \Delta G^{\circ}_{ads}}{T}. \quad (13)$$

The positive value of ΔS°_{ads} in the presence of inhibitor is an indication of increase in solvent entropy. It also interpreted with increase of disorders due to more water molecules which can be desorbed from the metal surface by one inhibitor molecule. Therefore, it is revealed that decrease in the enthalpy is the driving force for the adsorption of the inhibitor on the surface of the metal [37–39].

3.4. Mechanism of Corrosion Inhibition. The adsorption of the inhibitor molecule is often a displacement reaction involving removal of adsorbed water molecules from the metal surface,

$$\text{Inhibitor}_{(sol)} + x\text{H}_2\text{O}_{(ads)} \rightleftarrows \text{Inhibitor}_{(ads)} + x\text{H}_2\text{O}_{(sol)}, \quad (14)$$

In general, the physisorption involves electrostatic force of attraction between electrically charged surface of metal and charged species in the bulk of this solution. While

chemisorptions involve charge sharing or electrons transfer from the inhibitor molecule to the metal surface to form coordinate type of bond.

In HCl medium, the metal surface is negatively charged due to the specifically adsorbed Cl^- ions on the metal surface. In acidic solution, the nitrogen atom of the imidazole ring of the inhibitor can be protonated easily, due to high electron density on it, leading to positively charged inhibitor species. The adsorption can occur via electrostatic interaction between positively charged inhibitor molecules and negatively charged metal surface [11, 24]. Further, co-ordinate bond may be formed between unshared e^- pairs of unprotonated oxygen atom or nitrogen atom of the inhibitor and vacant d-orbital of mild steel surface. Additionally, inhibitor molecule may be chemically adsorbed due to interaction of π electrons of the inhibitor with metal surface [34].

4. Conclusion

(1) Miconazole nitrate is a good inhibitor for the corrosion of mild steel in 1 M HCl and inhibition efficiency was more pronounced with increase in the inhibitor concentration.

(2) The results obtained from polarization, impedance techniques, and weight loss measurements are in good agreement.

(3) The potentiodynamic polarization curves imply that the corrosion inhibition by miconazole nitrate for mild steel in 1 M HCl is mainly due to the suppression of anodic reaction.

(4) The inhibition efficiency of the inhibitor slightly increased with temperature for corrosion of mild steel.

(5) The adsorption of inhibitor on the surface of mild steel obeys Langmuir adsorption isotherm.

Acknowledgment

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