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Electrochemical Investigation of Meloxicam Drug as a Corrosion Inhibitor for Mild Steel in Hydrochloric and Sulfuric Acid Solutions

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ABSTRACT

Inhibition effects of meloxicam drug on mild steel corrosion in 1 M HCl and 0.5 M H_2SO_4 *solutions have been investigated by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. The inhibition efficiency (IE) was found to increase with increasing the inhibitor concentration in HCl solution. However, the IE was increased in* H_2SO_4 *solution with increasing the meloxicam concentration up to 200 ppm and then it was decreased. Maximum inhibition efficiency of 92 and 95% has been achieved at 400 and 200 ppm meloxicam in 1M HCl and 0.5M* H_2SO_4*, respectively. Adsorption of meloxicam on the surface of mild steel follows Langmuir isotherm. Potentiodynamic polarization measurements indicated that meloxicam is a mixed-type inhibitor. The effect of temperature on the corrosion rate of mild steel was studied in acidic media both in the absence and presence of meloxicam drug. Some thermodynamic parameters were computed from the effect of temperature on the inhibition process. Prog. Color Colorants Coat. 13 (2020), 155-165© Institute for Color Science and Technology.*

1. Introduction

Corrosion inhibitors are substances that decrease or prevent the reaction of the metal with the media when added in small concentrations to the corrosive media. The molecular structure of the most popular corrosion inhibitors contain S, N, O and P atoms and aromatic rings [1]. Although many organic inhibitors demonstrate high inhibition efficiency, they are mostly toxic and harmful to the environment [2].

Recently, researchers have paid attention to the development of non-toxic and green corrosion inhibitors, such as plant extracts and drugs to obviate the harmful effects of chemicals on the environment [3-13]. Because of their non-toxic characteristics and negligible negative impacts on the aquatic environment, drugs seem to be the ideal candidates to replace traditional toxic corrosion inhibitors. In 2019, the effect of penicillin G benzathine drug as a green corrosion inhibitor on mild steel in 1.0 M hydrochloric acid solution was investigated using potentiodynamic polarization and electrochemical impedance spectroscopy techniques [8]. Golestani et al. investigated the effect of penicillin G, ampicillin and amoxicillin drugs on the corrosion behavior of carbon steel in 1.0 M HCl solution using potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and electrochemical noise techniques [9]. There was the case of mixed mode adsorption here; penicillin was adsorbed mainly through chemisorption, while the two other drugs were adsorbed mainly through physisorption.

Soltaninejad et al. found that penicillin G showed the best inhibition effect on mild steel at 10 mM concentration in 3.0 M H_3PO_4 solution using

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potentiodynamic polarization and EIS techniques [10].

The effect of irbesartan drug as a corrosion inhibitor for mild steel in 1 M HCl and 0.5 M H_2SO_4 solutions has been investigated by open circuit potential curves, Tafel and linear polarization, and EIS techniques [11]. Maximum corrosion inhibition of 94 and 83% was obtained at 300 ppm irbesartan in 1 M HCl and 0.5 M H_2SO_4, respectively. Tasic et al. have investigated the effect of cephradine drug on the corrosion behavior of copper in 0.9% NaCl solution using EIS and potentiodynamic polarization methods [12]. Kumar et al. have shown that the traces of a semisynthetic antibiotic, cloxacillin, inhibits the corrosion of mild steel in acidic medium [13]. They have evaluated the inhibitory action of cloxacillin via weight loss, Tafel polarization, electrochemical impedance spectroscopy, and hydrogen permeation studies.

In this paper, meloxicam drug was investigated as a corrosion inhibitor for mild steel in 1.0 M HCl and 0.5 M H_2SO_4 solutions by Tafel polarization and electrochemical impedance spectroscopy techniques.

2. Experimental

2.1. Materials

The antibacterial drug, named meloxicam, was supplied from Sigma Aldrich and used without further purification. The chemical structure of the antibacterial drug is shown in Figure 1. The working electrode used here is made of mild steel (Ck45) with the surface area of 100 mm^2.

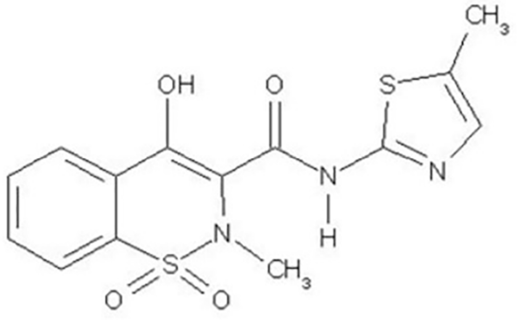

Figure 1: Structure of meloxicam.

2.2. Methods

EIS and potentiodynamic polarization measurements were used to investigate the corrosion rate of mild steel in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of meloxicam drug with different concentrations. Before performing the tests, the surface of the sample was rubbed with wet sandpapers through different grades then washed with distilled water and at last dried in air. The sample, as the working electrode (WE), was attached to a copper wire at one side and sealed by epoxy resin at another side.

A potentiostat/galvanostat Autolab 302N (Eco Chemie, Netherlands) supported by a frequency response analyzer FRA-2 and Nova 1.9 software was used for Potentiodynamic polarization and EIS tests. The counter electrode (CE) was prepared from a platinum rod and reference electrode was a saturated (KCl) Ag/AgCl electrode. The electrochemical tests order was EIS and afterward polarization technique. Before performing the tests, the specimens were immersed in the solution for about 30 min to obtain the stabilized open circuit potential (OCP).

For the EIS measurements, a sinusoidal potential signal of 10 mV (vs OCP) was used in the frequency range of 100 kHz-10 mHz. Nyquist plots of EIS data were analyzed by Nova 1.9 software. Tafel curves were recorded at a scan rate of 1 mV/s and Nova 1.9 software was used to determine the corrosion current density and polarization parameters.

3. Results and Discussion

3.1. Potentiodynamic polarization

Figure 2 shows the Tafel plots of mild steel exposed to 1.0 M HCl and 0.5M H_2SO_4 solutions in the absence and presence of different concentrations of meloxicam. It is obvious that the cathodic branch displays a typical Tafel behavior. As will be discussed later, this makes it possible to make an accurate evaluation of the cathodic Tafel slope (β_c) as well as corrosion currents (j_{corr}) by the Tafel extrapolation method. On the other hand, the anodic polarization curve does not display the expected log/linear Tafel behavior over the complete applied potential range. The curvature of the anodic branch may be attributed to the deposition of corrosion products or impurities (e.g. Fe_3C) on the steel surface to form a non-passive surface film [14]. Therefore, due to the nonlinearity in the anodic branch, it is impossible to obtain an accurate evaluation of the anodic Tafel slope by Tafel extrapolation of the anodic branch.

It has been shown that in the Tafel extrapolation method, the use of both anodic and cathodic Tafel regions is undoubtedly preferred over the use of only one Tafel region [15]. However, the corrosion rate can also be determined by Tafel extrapolation of either the cathodic or anodic polarization curve. In this case, the cathodic polarization curve is generally used which usually produces a longer and better defined Tafel region (as in our case).

For the calculation of anodic current density from the experimental data, the Tafel line of the cathodic polarization curve was first extended back to zero overvoltage, and then the anodic current density was calculated using equation (Eq. 1) [15].

$$i_a(\text{net experimental}) = i_a - |i_c| \quad (1)$$

where the subscripts *a* and *c* refer to the anodic and cathodic directions, respectively. Thus, the anodic current density is the sum of the experimentally observed anodic current density and the extrapolated cathodic current density.

Corrosion current density (i_{corr}), corrosion potential (E_{corr}), anodic and cathodic Tafel slopes (β_a and β_c) are listed in Table 1. The change in the values of β_c in the presence of inhibitor clearly indicates the effect of meloxicam on the kinetics of hydrogen evolution. The shift in the anodic Tafel slope (β_a) values may be due to the adsorption of inhibitor molecules onto the mild steel surface [16]. Therefore, the addition of inhibitor to acidic media affects both cathodic and anodic branches of the potentiodynamic polarization curves; it behaves as a mixed inhibitor.

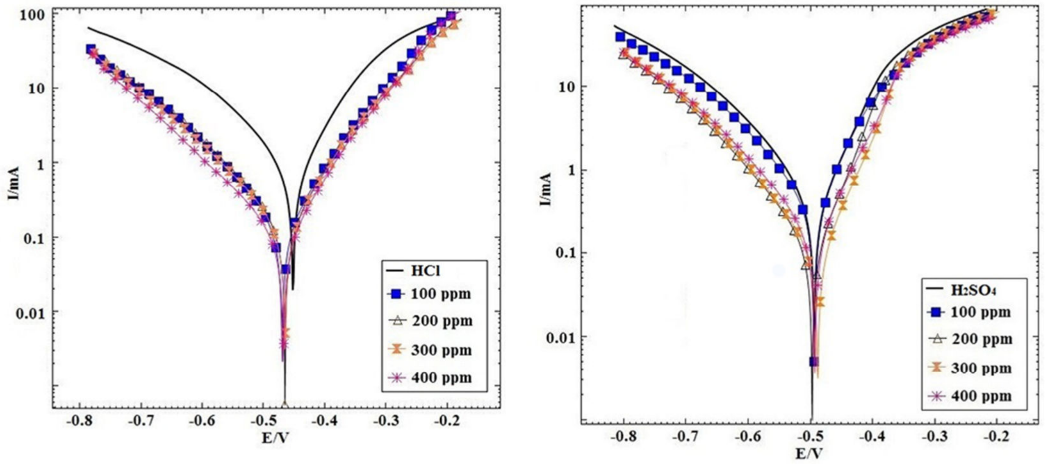

Figure 2: Polarization curves for mild steel in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of different concentrations of meloxicam at 25°C.

Table 1: Polarization parameters and the corresponding inhibition efficiencies for mild steel in 1.0 M HCl and 0.5 M H_2SO_4 containing different concentrations of meloxicam drug at 25 ^{0}C.

Solution	C /ppm	i_{corr}/µA.cm^{-2}	-E_{corr}/mV	β_a/mV.decade^{-1}	β_c/mV.decade^{-1}	IE_P(%)
HCl	0	1318	452	103	154	-
	100	188	470	95	127	85.7
	200	175	466	94	122	86.7
	300	158	465	89	129	88.0
	400	103	468	84	125	92.2
H_2SO_4	0	2557	495	148	230	-
	100	375	495	90	154	85.3
	200	142	498	59	124	94.4
	300	152	489	64	121	94.0
	400	204	489	62	117	92.0

Generally, if the shift in the absolute value of E_{corr} is more than 85 mV with respect to E_{corr} of the blank, the inhibitor can be considered as cathodic or anodic and if the displacement is less than 85 mV, the inhibitor can be considered as a mixed type [17, 18]. In the present work, the maximum change in equilibrium corrosion potential was -18 and +6 mV in HCl and H_2SO_4, respectively, suggesting that the meloxicam has acted as a mixed inhibitor which is further evidenced from shifting of both cathodic and anodic Tafel slopes. The corrosion current density decreased by increasing the inhibitor concentration to 400 ppm in HCl solution and to 200 ppm in 0.5 M H_2SO_4 solution.

Table 1 shows the values of corrosion inhibition efficiency (IE) expressed by the following equation (Eq. 2) [19].

$$IE_P(\%) = \frac{i_{corr} - i'_{corr}}{i_{corr}} \times 100 \tag{2}$$

where i_{corr} and i'_{corr} are corrosion current densities in the uninhibited and inhibited solutions, respectively. IE_P values increased with increasing the meloxicam concentration and reached its maximum value for 400 ppm and 200 ppm of meloxicam in HCl and H_2SO_4 solutions, respectively. These values also indicate that the drug acts as an effective inhibitor.

Since the inhibition efficiency is due to the blocking effect of the adsorbed species, the surface coverage, θ, can be calculated by θ=IE(%)/100. The adsorption isotherms of Langmuir, Temkin, and Frumkin were used for fitting data and the best results were found for Langmuir isotherm (Figure 3), which can be expressed in the following way (Eq. 3).

$$\frac{C}{\theta} = C + \frac{1}{K} \tag{3}$$

where θ is the surface coverage, C is the inhibitor concentration and K is the adsorption equilibrium constant. As can be seen in Figure 3, the plot of C/θ vs. C for the inhibitor gives straight lines with correlation coefficients and slopes close to 1 which confirms that the adsorption of the inhibitor is well described by the Langmuir adsorption isotherm. The calculated adsorption coefficient, K_{ads}, was higher in H_2SO_4 than in HCl, meaning that the adsorption on active sites of steel surface for inhibitor molecules was easier in H_2SO_4 solution than in HCl solution [11].

3.2. Electrochemical impedance spectroscopy

The results of EIS measurements for mild steel in both HCl and H_2SO_4 solutions in the absence and presence of meloxicam are shown in Figure 4. The investigated electrochemical system has resistive and capacitive elements since the Nyquist plots are in the form of semi-circles. The electrochemical system showed modified capacitive behavior in both acid solutions due to the lack of a perfect semi-circle in the Nyquist plots. According to Nyquist plots, the charge transfer resistance (R_{ct}) increased with meloxicam concentration and reached its maximum value for 400 and 200 ppm of meloxicam in HCl and H_2SO_4 solutions, respectively.

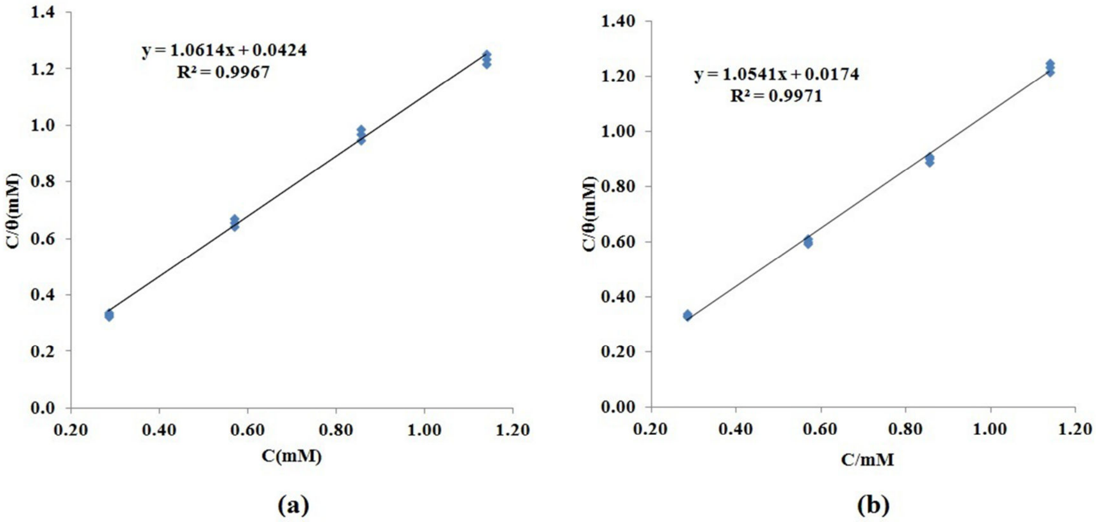

Figure 3: Langmuir adsorption isotherm of the inhibitor calculated by Tafel polarization data for mild steel in (a) 1.0 M HCl and (b) 0.5 M H_2SO_4 solutions.

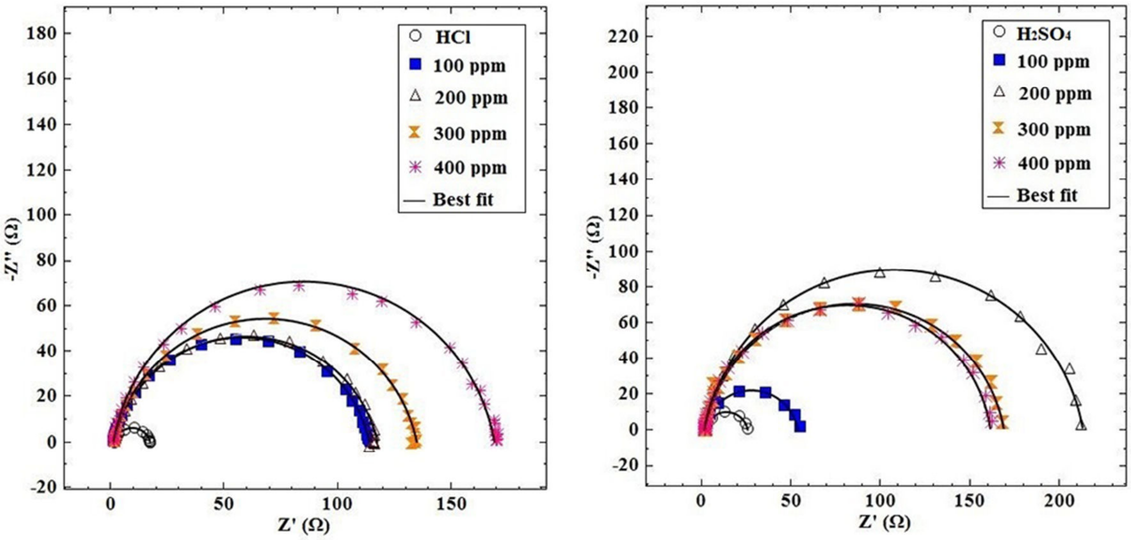

Figure 4: Nyquist plots for mild steel in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of different concentrations of meloxicam at 25°C.

For investigating the details of electrochemical reactions occurring at the electrode/solution interface, an equivalent electrochemical circuit was used to fit the EIS results as shown in Figure 5. The corrosion process can be represented by constant phase element (CPE) and charge transfer resistance R_{ct} in parallel. The impedance value (Z_{CPE}) of CPE can be determined by the following equation (Eq. 4) [20].

$$Z_{CPE} = \frac{1}{Y_0 (j\omega)^n} \tag{4}$$

where Y_0 is the CPE constant, ω is angular frequency and n is the CPE exponent. Table 2 lists the impedance parameters obtained from the fitting of Nyquist plots with the proposed equivalent circuit using Nova1.9 software.

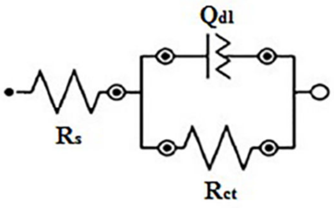

Figure 5: The equivalent circuit used to fit the experimental data.

It is evident that R_{ct} values increased by increasing the concentration of inhibitor. This condition is due to the increasing of the surface coverage by the inhibitor, which can lead to higher inhibition efficiency. The values of IE in Table 2 were computed using the following equation (Eq. 5):

$$IE_{EIS}(\%) = \frac{R'_{ct} - R_{ct}}{R'_{ct}} \times 100 \tag{5}$$

where R_{ct} and R'_{ct} are the charge transfer resistance before and after addition of the inhibitor to the corrosive media, respectively. IE values increased as the concentration of inhibitor increased but there is a restriction in the sense that an additional increase in the inhibitor concentration did not make any considerable change in the inhibition efficiency of the inhibitor. The IE values acquired from the EIS method (Table 2) are completely consistent with those acquired from the polarization method and actually confirm each other (Table 1). As can be seen from Figure 6, the EIS results are compatible with the Langmuir isotherm.

Table 2: Impedance parameters and the corresponding inhibition efficiency values for mild steel in 1.0 M HCl and 0.5 M H_2SO_4 containing different concentrations of meloxicam drug at 25 °C.

Solution	C /ppm	$R_s/\Omega.cm^2$	$R_{ct}/\Omega.cm^2$	n	10^6 $Y_0/F.cm^{-2}.s^{n-1}$	$IE_{EIS}(\%)$
HCl	0	1.5	15.8	0.872	307	-
	100	1.0	113	0.874	62.2	86.0
	200	1. 3	116	0.861	79	86.4
	300	1.3	133	0.872	64.9	88.1
	400	1.0	168	0.891	47.1	90.6
H_2SO_4	0	1.8	9.3	0.906	230	-
	100	1.8	52	0.905	101	82.1
	200	2.0	211	0.898	46	95.6
	300	2.0	167	0.895	57	94.4
	400	1.6	160	0.916	50	94.2

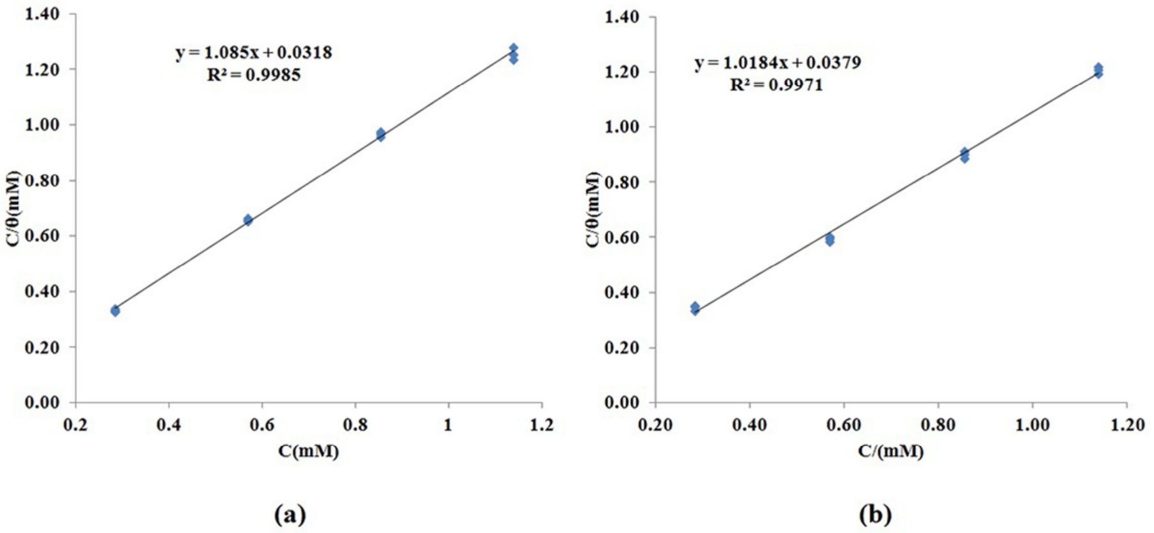

Figure 6: Langmuir adsorption isotherm of the inhibitor calculated by EIS data for mild steel in (a) 1.0 M HCl and (b) 0.5 M H_2SO_4 solutions.

3.3. Temperature effect and thermodynamic parameters

Potentialdynamic polarization measurements in the range of 25-55 °C in the absence and presence of different concentrations of drug were used to investigate the adsorption of inhibitor and determine the E_a and thermodynamic parameters of the mild steel corrosion process in acidic solutions. The Tafel curves for mild steel in both acidic solutions in the absence and presence of the optimum concentrations of the drug are demonstrated in Figures 7 and 8. Corrosion factors at various temperatures are listed in Tables 3 and 4. The results acquired from the polarization curves proved an increase in i_{corr} and decrease in IE% with increasing the temperature. In general, the reason for reducing the inhibition efficiency that occurs as a result of rising temperature can be expressed as follows. The increase in temperature reduces the time lag between the adsorption and desorption of drug molecules on the surface of mild steel [21]. Therefore, the mild steel surface experiences longer exposure to the acidic media, resulting in an increase in corrosion rate with increasing the temperature, after which the IE% values fall at higher temperatures.

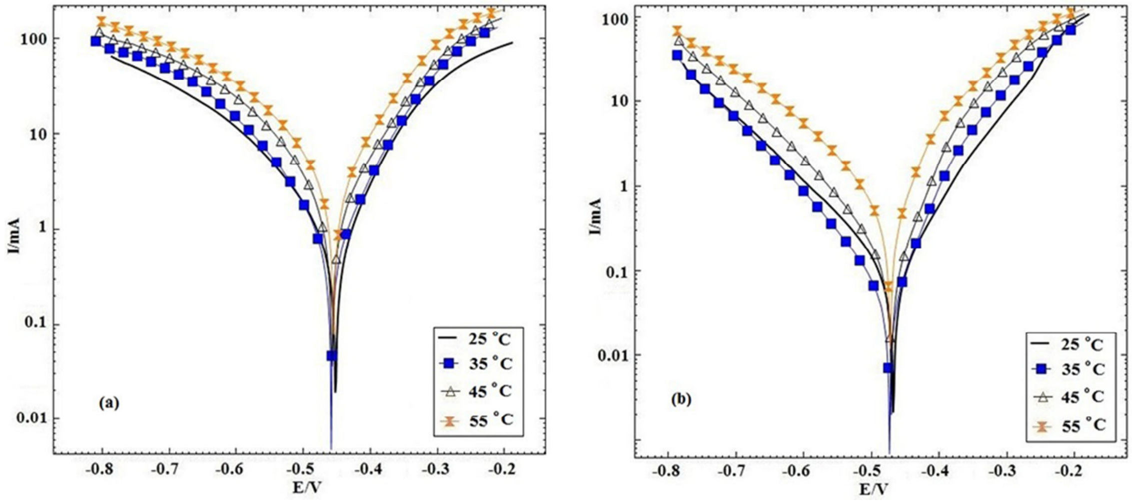

Figure 7: Effect of temperature on the polarization curves in 1.0 M HCl solution (a) without inhibitor and (b) in the presence of 400 ppm meloxicam.

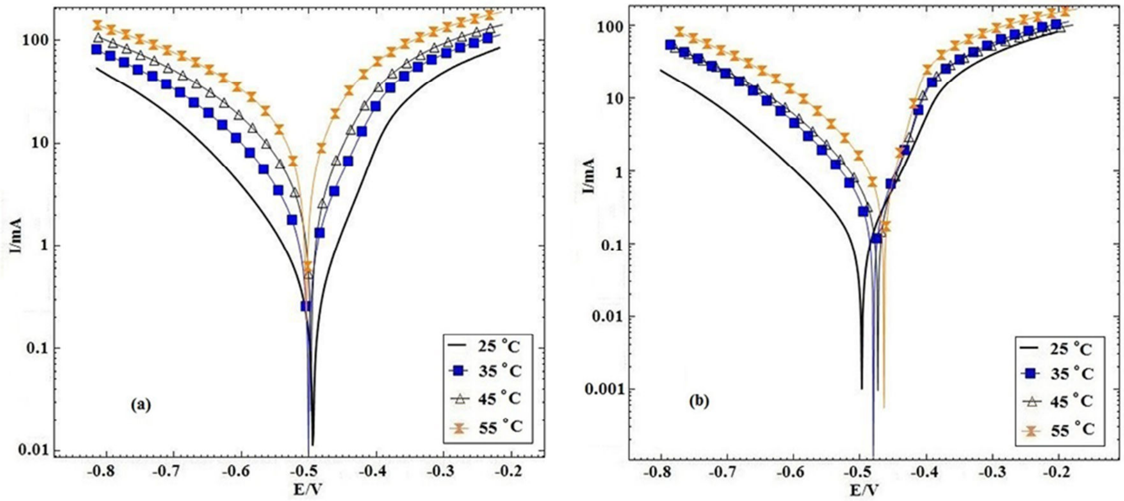

Figure 8: Effect of temperature on the polarization curves in 0.5 M H_2SO_4 solution (a) without inhibitor and (b) in the presence of 200 ppm meloxicam.

Table 3: Effect of temperature on the corrosion parameters of mild steel in 1.0 M HCl solution containing meloxicam drug.

Drug/ppm	T/°C	i_{corr}/ µA.cm^{-2}	-E_{corr}/mV	IE%
0	25	1318	452	-
	35	2214	455	-
	45	3345	457	-
	55	6094	451	-
400	25	103	468	92.2
	35	205	463	90.7
	45	454	470	86.4
	55	1085	471	82.2

Table 4: Effect of temperature on the corrosion parameters of mild steel in 0.5 M H_2SO_4 solution containing meloxicam drug.

Drug/ppm	T/°C	i_{corr}/ µA.cm^{-2}	-E_{corr}/mV	IE%
0	25	2557	495	-
	35	4652	487	-
	45	9243	486	-
	55	19162	486	-
200	25	135	498	94.7
	35	562	491	87.9
	45	1037	486	88.8
	55	3215	490	83.2

Arrhenius equation (Eq. 6) can express the dependence of the corrosion rate on temperature.

$$i_{corr} = A\exp\left(\frac{-E_a}{RT}\right) \qquad (6)$$

Where i_{corr} is the corrosion current, A is constant, E_a is the activation energy, R is the gas constant and T is the absolute temperature. E_a values can be obtained from the slope of Arrhenius plot [lni_{corr}vs 1/T]. Figures 9 and 10 show Arrhenius plots for mild steel electrode in 1 M HCl and 0.5 MH_2SO_4 solutions in the absence and presence of meloxicam. A careful analysis of Figures 9 and 10 revealed that the calculated E_a values obtained for HCl and H_2SO_4 solutions in the absence of meloxicam were 40.6 and 54.6 kJ/mol, respectively, which were smaller than those in the presence of meloxicam (63.7 and 82.4 kJ/mol) (Table 5). This fact indicated that the corrosion rate of mild steel in acidic solutions decreased by meloxicam drug [9].

Reducing the inhibition efficiency with increasing the temperature and a similar increase in the E_a value in the presence of inhibitor can be interpreted as the formation of physical adsorption film on the surface of the alloy [22, 23]. The value of adsorption equilibrium constant, K_{ads}, can be calculated by the reciprocal of the intercept of the isotherm line (Figure 3). The free energy of inhibitor adsorption on mild steel surface is obtained from the following equation (Eq. 7),

$$\Delta G_{ads} = -RT\ln(55.5K_{ads}) \qquad (7)$$

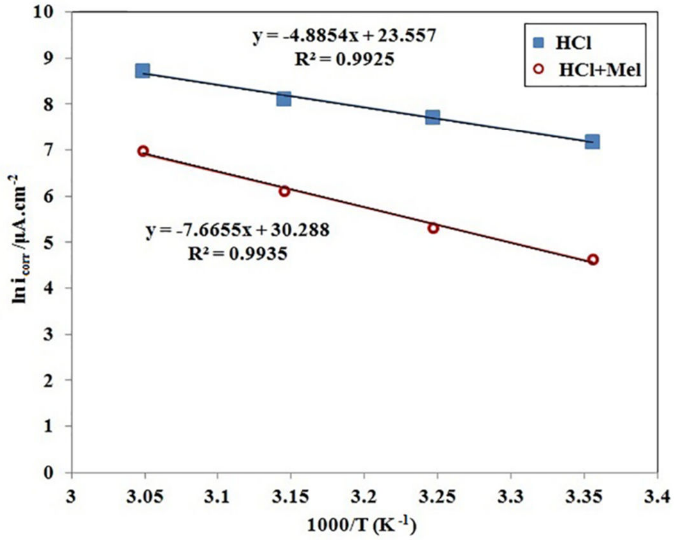

Figure 9: Arrhenius plots for mild steel in 1.0 M HCl solution in the absence and presence of 400 ppm meloxicam.

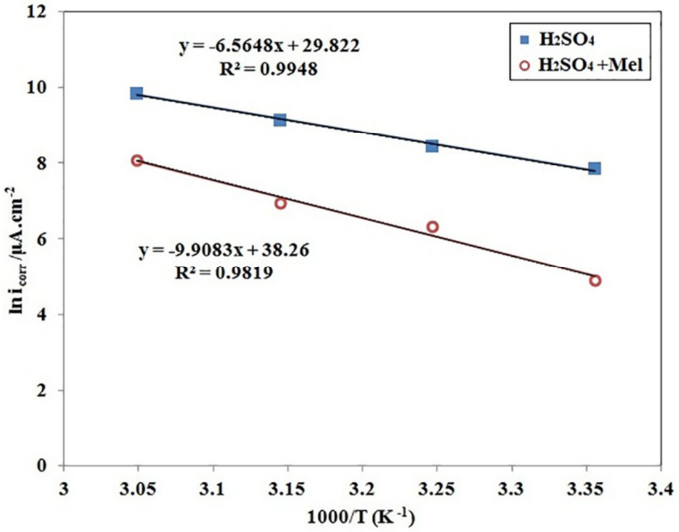

Figure 10: Arrhenius plots for mild steel in 0.5 M H_2SO_4 solution in the absence and presence of 200 ppm meloxicam.

where 55.5 is the molar concentration of water in the solution, R is the gas constant (8.314 J K^{-1}.mol^{-1}) and T is the absolute temperature (K). The values of K_{ads} and ΔG_{ads} are summarized in Table 5. An acceptable conformity was found between the two techniques. Generally, if ΔG_{ads} values are more positive than −20 kJ/mol, it can be said that physical adsorption has occurred and if ΔG_{ads} values are more negative than −40 kJ mol^{-1}, it can be said that chemical adsorption has occurred. Therefore, it can be concluded from the obtained value for ΔG_{ads} that the adsorption of meloxicam is not solely chemisorption or physisorption but involving comprehensive adsorption (both chemical and physical).

Langmuir adsorption isotherm is expressed by the following equation (Eq. 8) [24].

$$\ln\left(\frac{\theta}{1-\theta}\right) = \ln A + \ln C - \frac{\Delta H_{ads}}{RT} \qquad (8)$$

where θ is the surface coverage, A is independent constant, C is the concentration, R is the gas constant, T is the absolute temperature, and ΔH_{ads} is the adsorption enthalpy.

The plot of ln(θ/(1-θ)) versus 1/T at constant additive concentration gives a straight line as shown in Figure 11. The slope of the straight line is $-\Delta H_{ads}/R$. The calculated value of ΔH_{ads} for inhibitor adsorption is given in Table 5. The negative values of ΔH_{ads} represent the exothermic behavior of inhibitor on the mild steel surface. In an exothermic process, the absolute value of the adsorption enthalpy can distinguish the physisorption from chemisorption. In general, the values of ΔH_{ads} around 40 kJ mol^{-1} or less are consistent with physisorption process, and those around 100 kJ mol^{-1} indicate chemisorption process the absolute enthalpy of a physisorption process is lower than 40 kJ.mol^{-1} [25]. In this paper, the value of enthalpy is larger than the usual physical adsorption heat and is smaller than the chemical adsorption heat. Then, it can be concluded that the adsorption was carried out both physically and chemically. The adsorption entropy (ΔS_{ads}) can be calculated using the following equation (Eq. 9):

$$\Delta G_{ads} = \Delta H_{ads} - T\Delta S_{ads} \qquad (9)$$

The obtained values for ΔS_{ads} are shown in Table 5. The positive values of ΔS_{ads} can be explained as follows. The adsorption of inhibitor molecules from the aqueous solution is a substitution process between the inhibitor molecules in the aqueous phase and water molecules on the alloy surface [16]. The adsorption of inhibitor molecules on the electrode surface is accompanied by desorption of water molecules from the electrode surface. Thus, while the adsorption process for the inhibitor is associated with a decrease in entropy, the opposite is true for the solvent. Thermodynamic values are the algebraic sum of the adsorption of inhibitor molecules and the desorption of water molecules [26]. The positive values of ΔS_{ads} suggest an increase in the disordering of reactants with respect to the alloy/solution interface.

Table 5: Activation and thermodynamic parameters of adsorption obtained by potentiodynamic polarization measurements for mild steel in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of the optimum concentration of meloxicam drug.

Sample	E_a (kJ.mol^{-1})	K_{ads} (M^{-1})	ΔG_{ads} (kJ.mol^{-1})	ΔH_{ads} (kJ.mol^{-1})	ΔS_{ads} (J.K^{-1}. mol^{-1})
HCl	40.6	-	-	-	-
Mel+HCl	63.7	23256	34.9	-25.7	31.1
H_2SO_4	54.6	-	-	-	-
Mel+H_2SO_4	82.4	59880	37.2	-34.0	10.9

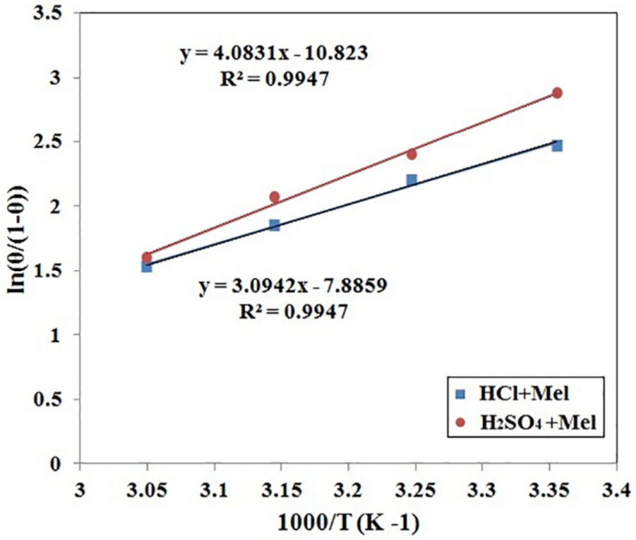

Figure 11: Plot of ln (θ/1–θ) vs. 1/T for mild steel in1.0 M HCl and 0.5 M H_2SO_4 solutions containing 400 ppm and 200 ppm meloxicam, respectively.

4. Conclusion

Meloxicam drug was tested as a corrosion inhibitor for mild steel in 1 M HCl and 0.5 M H_2SO_4 solutions. Tafel polarization curves showed that the inhibitor retarded the corrosion of mild steel by lowering the corrosion current. According to EIS measurements, the corrosion inhibition was caused by an increase in the charge transfer resistance of mild steel at the metal-acid interface. The data acquired from the potentiodynamic polarization and EIS measurements indicated that the adsorption of meloxicam on mild steel in both HCl and H_2SO_4 solutions follows the Langmuir isotherm. The potentiodynamic polarization experiments at different temperatures suggested that meloxicam can effectively minimize the corrosion effects of both acidic solutions on the mild steel surface. The reason for inhibition of mild steel in both acidic media by meloxicam was due to the adsorption of meloxicam molecules on the alloy surface which was also evidenced by Langmuir isotherm study. The IE and ΔG_{ads} values obtained from Tafel method showed an acceptable compatibility with the data acquired from the EIS technique.

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