



Electrochemical and Surface Investigation of *Quinoline-8-sulphonyl chloride* as Corrosion Inhibitor for Mild Steel in Acidic Medium

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Abstract: The primary objective of this study is to examine the corrosion inhibition performance of quinoline-8-sulphonyl chloride (QSC) on Mild Steel in 1 M H₂SO₄ solution by electrochemical, weight loss, Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray spectroscopy (EDX) methods. From the Weight Loss (WL) method, it is observed that the corrosion rate has been decreasing with the addition of inhibitor concentration, which indicates that, the formation of protective film on the surface of mild steel. Electrochemical studies showed that QSC is a good corrosion inhibitor for mild steel in acid solution. Tafel plots indicate that QSC is a mixed inhibitor. The data, which is obtained from all methods, show that the adsorption of the used inhibitor obeys Langmuir adsorption isotherm. SEM and EDX techniques confirmed the formation of protective film on the surface of mild steel.

1. Introduction

The corrosion of the metal and its alloys is a common problem with economic implications costing billions of dollars in every year. The protection of metals and its alloys against corrosion the use of corrosion inhibitor is one of the most economical and practical methods (1-3). Corrosion inhibitors have been widely used in many industries to reduce the corrosion rate of metal surfaces in contact with aggressive medium (4-6). Corrosion inhibitors are organic or inorganic or mixed material substances, which decreases the rate of corrosion (7). The addition of inhibitor effectively secures the metal and its alloys against an acid attack. The majority of the well-known corrosion inhibitors are organic compounds containing heteroatom such as S, N and O with multiple bonds, which allow an adsorption on the metal surface (8, 9). These compounds are adsorbed on the metal surface by physically or chemically blocking the active sites of corrosion. The adsorption may occur in (i) electrostatic attraction between the charged inhibitor molecule and the charged metal, (ii) interaction between the electron pairs in the inhibitor molecule and the metal, (iii) π electron interaction with the metal and (iv) a combination of all (10, 11). The further reference of literature reveals that quinoline and some of its derivatives such as quinaldine, quinaldic acid (12), 4-hydroxy quinoline-2-carboxylic acid (7), 8-hydroxy quinoline, 3-formyl-8-hydroxy quinoline (13), 2-chloro quinoline 3-carbaldehyde, (2-chloro-quinoline-3-ylmethylene)-*p*-tolyl-amine (14) are used as an effective corrosion inhibitors used for the mild steel in acidic media. In this existing research, the inhibition performance of QSC on mild steel in 1 M sulphuric acid solution has been investigated and the results are discussed by the electrochemical and surface morphological studies.

2. Experimental Procedure

2.1. Materials Preparation

The corrosion inhibition tests are carried out on rectangular mild steel specimens with the chemical composition of (% wt) 0.104 % C, 0.58 % Mn, 0.035 % P, 0.026 % S and the rest Fe, which was mechanically cut into 3.5 x 1.5 x 0.2 cm dimensions for weight loss measurements and 1.0 x 1.0 x 0.2 cm dimensions for surface investigation. On behalf of an electrochemical measurement, 1.0 cm² surface area of the material is exposed to the corrosive medium and the balance being covered through commercially available epoxy resin. The exposed surface area of the mild steel specimens are polished the mirror to conclude with numerous grades of emery papers. Subsequently, it is cleaned with AR grade acetone and finally dried in air drier before immerse in the corrosive medium.

2.2. Corrosive Medium

The 1 M H₂SO₄ acid solution is prepared for analytical grade of 97 % H₂SO₄ and double-distilled water.

2.3. Inhibitor Preparation

The investigated heterocyclic compound namely *quinoline-8-sulphonyl chloride* is obtained from Sigma-Aldrich. The structure of the studied organic compound is given in **Figure 1**. The stock solution of studied compound (QSC) is made in 1 M H₂SO₄ solution by dissolving the compound in ethanol (1gm/50 ml) and it has been used for all the studies. The different concentrations of inhibitor solution like 50 to 300 ppm are prepared from the stock solution and 1 M H₂SO₄ solution alone is also taken for comparison of all experimental studies.

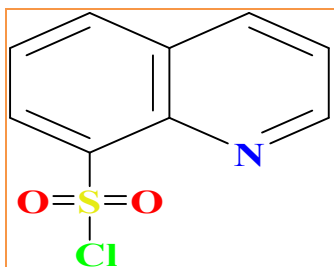


Figure 1: Molecular structure of QSC [IUPAC name: *Quinoline-8-sulphonyl chloride*]

2.4. Weight loss studies

The weight loss method is the most widely acceptable method of corrosion inhibition assessment (15). In this method, the metal specimens are immersed for 3 hrs in different concentrations of both inhibitor solution and blank solution. After the immersion hrs the surface of the mild steel specimens are cleaned by acetone and weighed accurately.

From the analysis, the corrosion rate (W), inhibition efficiency (IE %) and the degree of surface coverage (θ) is determined by the following equations:

$$\text{C.R.} = W = \frac{m_1 - m_2}{St} \quad (1)$$

where, m_1 is the mass of the polished mild steel, m_2 is the mass of the corroded mild steel, S is the surface area of the mild steel, t is the immersion time and W is the weight loss of the mild steel specimen.

$$\text{IE (\%)} = \left[\frac{W_o - W_i}{W_o} \right] \times 100 \quad (2)$$

$$\theta = \left[\frac{W_o - W_i}{W_o} \right] \quad (3)$$

where, W_o is the rate of corrosion in the absence of QSC and W_i is the rate of corrosion in the presence of QSC, respectively.

2.5. Electrochemical studies

The electrochemical studies of inhibitors have the most important benefits in short measurements time and giving vital information about the mechanism in corrosion inhibition. All the electrochemical studies namely Electrochemical Impedance Spectroscopy (EIS), Potentiodynamic polarization (Tafel plots) and Linear Polarization Resistance (LPR) are carried out using CH-Electrochemical analyzer model 760 D with CHI 760 D software. A conventional three-electrode system is used for both polarization (potentiodynamic polarization and linear polarization) and EIS studies. In this setup, the polished, cleaned and dried mild steel with 1.0 cm² exposed area is used as working electrode. In further, platinum electrode is used as an auxiliary electrode and saturated calomel electrode is used as reference electrode. Before starting the electrochemical studies, the test sample (mild steel) is allowed to reach steady-state value of Open Circuit Potential (OCP). All the three electrodes have immersed in 1 M H₂SO₄ solution in both the absence and presence of different concentrations of inhibitor. The above set up has been kept in room temperature for 30 mins and then electrochemical studies have been performed.

2.6. Electrochemical Impedance Spectroscopy (EIS) studies

EIS studies are measured in the frequency range of 10,000 Hz to 0.1 Hz with ac impedance signal of 0.01 V amplitude. From this analysis, Nyquist and Bode plots are obtained. From Nyquist plots, the corrosion parameters such as R_{ct} , Y_{max} and C_{dl} of mild steel in absence and presence of different concentrations of QSC in 1 M H₂SO₄ solutions are computed. The inhibition efficiencies are calculated by using R_{ct} values as in the following equation:

$$IE (\%) = \left[\frac{R_{ct}^i - R_{ct}^o}{R_{ct}^i} \right] \times 100 \quad (4)$$

where, R_{ct}^i is the charge transfer resistance values of QSC contained solution and R_{ct}^o is the charge transfer resistance value of the blank solution (absence of QSC), respectively.

2.7. Potentiodynamic polarization studies

The potentiodynamic polarization studies are started from -300 mV cathodically to +300 mV anodically in respect to OCP with a scan rate of 0.1 mV/s. The experiments are carried out at room temperature and the corrosion parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), anodic slope (β_a) and cathodic slope (β_c) are obtained by Tafel curves. The inhibition efficiencies (IE %) have been evaluated from I_{corr} values using the following equation:

$$IE (\%) = \left[\frac{I_{corr}^o - I_{corr}^i}{I_{corr}^o} \right] \times 100 \quad (5)$$

where, I_{corr}^o is the corrosion current density without QSC and I_{corr}^i is the corrosion current density with QSC, respectively.

2.8. Linear Polarization Resistance (LPR) studies

In the linear polarization resistance studies, the potential of the electrode has been scanned from -0.02 to +0.02 V versus E_{corr} at a scan rate of 0.125 mV/s (16). The inhibition efficiency (IE %) and surface coverage (θ) is calculated from R_p values according to the following equations:

$$\theta = \left[\frac{R_p^i - R_p^o}{R_p^i} \right] \quad (6)$$

$$\text{IE (\%)} = \left[\frac{R_p^i - R_p^o}{R_p^i} \right] \times 100 \quad (7)$$

where, R_p^i and R_p^o are the linear polarization resistance values in the presence and absence of QSC, respectively.

2.9. Surface Investigation studies

2.9.1. Scanning Electron Microscopy (SEM) studies

The surface of the polished mild steel, corroded mild steel and inhibited mild steel are investigated by using JEOL/EO JSM-6390 model SEM.

2.9.2. Energy Dispersive analysis of X-rays (EDX) studies

EDX system attached with a JEOL/EO JSM-6390 scanning electron microscopy are used for elemental analysis of polished mild steel surface and film formed mild steel surface before (corroded MS) and after applying (inhibited) the studied inhibitor.

3. Results and Discussion

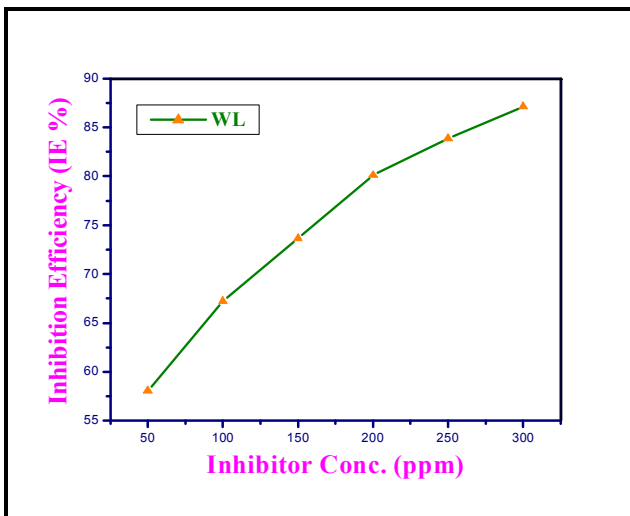
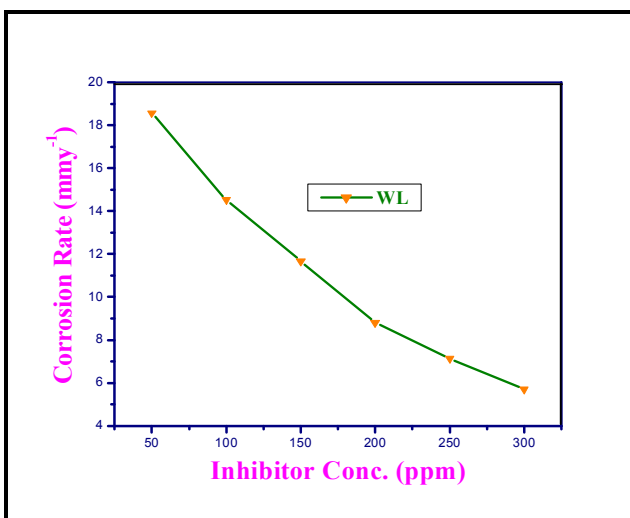
3.1. Weight loss studies

The values of result in the weight loss methods are reported in **Table 1** and the corrosion behavior of mild steel in same acidic media are shown in **Figure 2 & 3**. The percentage inhibition efficiency (IE %) and surface coverage (θ) is determined the above-mentioned equation.

The **Table 1** data reveals that, the inhibition efficiency increases, with increase in concentration of the investigated inhibitor. The increase in efficiency from 58.07 % to 87.11 % may be due to the blocking effect of the active sites of the metal surface by both adsorption and film formation mechanisms, which decreases the effective area of corrosion attack (17). **Figure 2** shows that the inhibition efficiency increases, with increasing concentration of inhibitor and the highest inhibition efficiency of 87.11 % is obtained at 300 ppm of QSC, which explains the effect of protection of mild steel against corrosion attack from the type of selected and studied inhibitor, because the inhibitor molecule contains benzene ring, number of heteroatom's and π electrons. In **Figure 3**, the corrosion rate of mild steel decreases from 18.57 mmy^{-1} to 5.71 mmy^{-1} with increasing concentration of inhibitor, this trend may result from the fact that adsorption and surface coverage of the metal increases with the addition of QSC. Thus, the surface is separated effectively from the corrosive medium. From this study, it is clear that the inhibitor is adsorbed at the corrosion sites of mild steel surface and responsible for corrosion resistance.

Table 1: Weight loss results of the mild steel corrosion without and with the addition of different concentrations of QSC in 1 M H₂SO₄ solution

<i>Concentration of the Inhibitor (ppm)</i>	<i>Weight loss (mg cm⁻²)</i>	<i>Corrosion rate (mm y⁻¹)</i>	<i>Surface coverage (θ)</i>	<i>IE (%) η</i>
Blank	18.6	44.29	-	-
50	7.8	18.57	0.5807	58.07
100	6.1	14.52	0.6722	67.22
150	4.9	11.67	0.7365	73.65
200	3.7	8.81	0.8011	80.11
250	3.0	7.14	0.8388	83.88
300	2.4	5.71	0.8711	87.11

**Figure 2: Plot of inhibition efficiency against different concentrations of QSC****Figure 3: Plot of corrosion rate against different concentrations of QSC**

3.2. Electrochemical Impedance Spectroscopy (EIS) studies – Nyquist and Bode plots

The corrosion inhibition behavior of mild steel in 1 M H₂SO₄ solution without and with QSC is investigated by EIS studies at room temperature. From these studies the obtained plots of Nyquist and bode are shown in **Figure 4 & 5**. The important impedance parameters are derived from Nyquist plots are given in **Table 2**. From the Nyquist plot (**Figure 4**), the R_{ct} values are determined from the difference in impedance at

lower and higher frequencies (18). The C_{dl} values are calculated at maximum frequency (f_{max}), at which the imaginary component of the Nyquist plot is maximum and calculated by using the following equation:

$$C_{dl} = (2\pi f_{max} R_{ct})^{-1} \tag{8}$$

where, C_{dl} is double layer capacitance, f_{max} is the maximum frequency and R_{ct} is the charge transfer resistance, respectively.

From *Figure 5*, the phase angle at high frequencies gives a general idea of the corrosion resistance of QSC. (The more negative of the phase angle the more capacitive of the electrochemical behavior (19). This is an indication of superior inhibitive behavior of inhibitor at high concentration. *Table 2* indicates that the R_{ct} values are increased ($2.008 \Omega \text{cm}^2$ to $10.549 \Omega \text{cm}^2$) and that the C_{dl} values are decreased ($78984.6 \mu\text{Fcm}^{-2}$ to $2865.3 \mu\text{Fcm}^{-2}$) with increasing inhibitor concentration. These results indicate a decrease in the active surface area caused by the adsorption of the inhibitors on the mild steel surface. And it suggests that the corrosion process become controlled. On the other hand, the decrease in this capacitor values can be attributed to the formation of a protective layer on the electrode-mild steel surface (20). The thickness of the protective film δ_{inh} is related to C_{dl} by the equation (21):

$$\delta_{inh} = \frac{\epsilon_0 \epsilon_r}{C_{dl}} \tag{9}$$

where, ϵ_0 is the dielectric constant and ϵ_r is the relative dielectric constant.

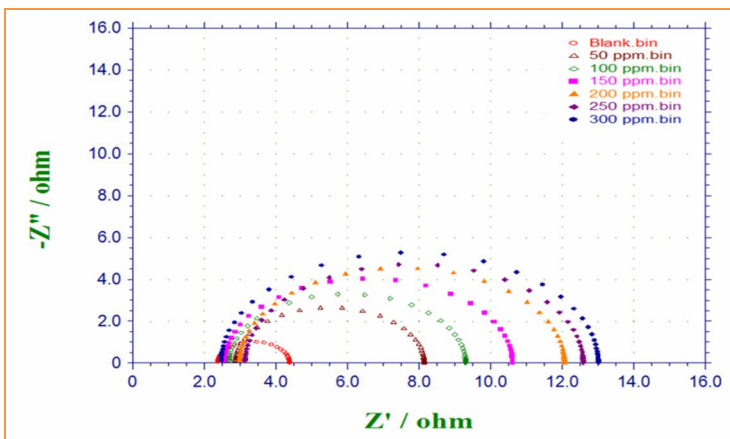


Figure 4: Nyquist plot of mild steel in 1 M H_2SO_4 solution with and without various concentrations of QSC

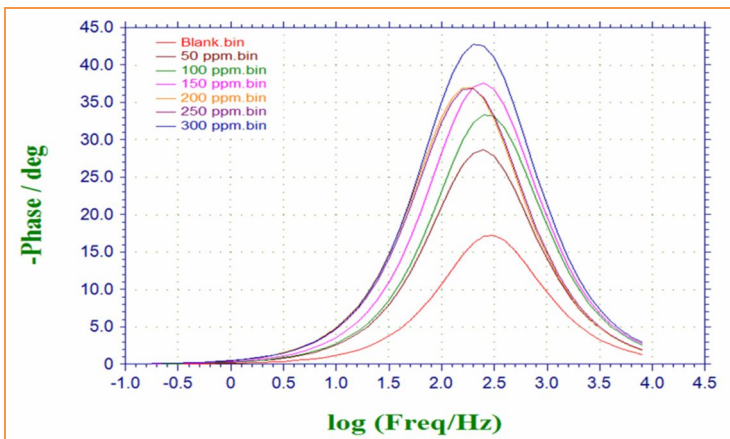


Figure 5: Bode plot of mild steel in 1 M H_2SO_4 solution with and without various concentrations of QSC

Table 2: Nyquist plots values for mild steel in 1 M H₂SO₄ solution containing various concentrations of QSC

Concentration of the Inhibitor (ppm)	Y_{max} (Ωcm^2)	R_{ct} (Ωcm^2)	C_{dl} ($\mu F cm^{-2}$)	Surface coverage (θ)	IE (%) η
Blank	1.004	2.008	78984.6	-	-
50	2.610	5.288	11537.4	0.6202	62.02
100	3.288	6.596	7342.2	0.6955	69.55
150	4.006	8.013	4960.6	0.7494	74.94
200	4.512	9.073	3889.7	0.7786	77.86
250	4.703	9.445	3584.7	0.7874	78.74
300	5.268	10.549	2865.3	0.8096	80.96

The *Table 2* confirms that the inhibition efficiency (IE %) increases with the concentrations of the investigated inhibitor and maximum efficiency (80.96%) reaches at optimum concentration (300 ppm) of QSC. All the above results reveal that, with increase in QSC concentration, the protective layer is highly protective.

3.3. Potentiodynamic polarization studies – Tafel Curves

The potentiodynamic polarization curves for mild steel in 1 M H₂SO₄ solution in absence and presence of various concentrations of QSC are shown in *Figure 6*. It could be noted that, the increase in the concentration of QSC alters both anodic and cathodic area reactions particularly in an optimum concentration (300 ppm). In the review of literature (22, 23), it has been reported that if the displacement of corrosion potential (E_{corr}) is greater than 85 mV with respect to the corrosion potential (E_{corr}) of the blank solution, the inhibitor can be seen as a cathodic or anodic type and if the displacement of corrosion potential (E_{corr}) is less than 85 mV, the inhibitor can be seen as mixed type. In the present study, the maximum displacement of corrosion potential (E_{corr}) is 77 mV cathodically with respect to the blank solution. Therefore, the investigated inhibitor could be classified into mixed-type with slightly cathodic in nature. The important electrochemical corrosion kinetic parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), anodic (β_a) and cathodic (β_c) Tafel plots and linear polarization resistance (R) derived from Tafel extrapolation of the polarization curves and all the parameter values including the inhibition efficiency (IE %) are given in *Table 3*. The percentage inhibition efficiency (IE %) are determined by using the above-mentioned equation. The results show that the corrosion current density (I_{corr}) values decrease with the addition of inhibitor concentrations, which explains the lower I_{corr} values of mild steel specimen was highly protected from the corrosion environment.

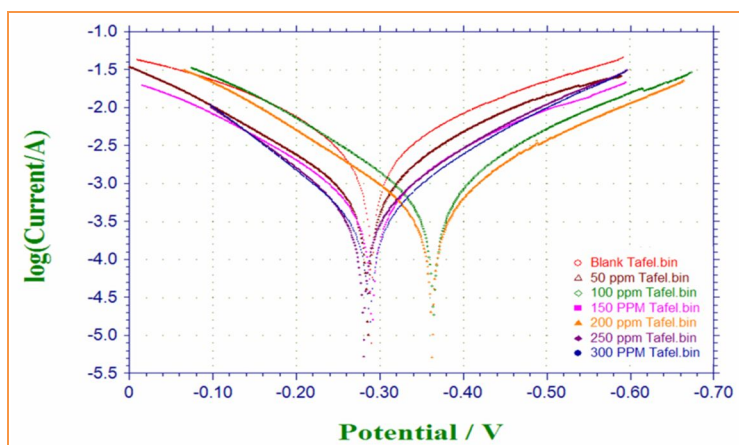
**Figure 6:** Tafel curves of mild steel in 1 M H₂SO₄ solution with and without various concentrations of QSC

Table 3: Tafel curves values for mild steel in 1 M H₂SO₄ solution containing various concentrations of QSC

Concentration of the Inhibitor (ppm)	β_a (V/dec)	β_c (V/dec)	E_{corr} (mV/SCE)	I_{corr} (mA/cm ²)	Surface coverage (θ)	IE (%) η
Blank	6.738	6.157	-292	2.195	-	-
50	6.805	7.074	-285	0.8076	0.6321	63.21
100	7.414	7.206	-358	0.6301	0.7129	71.29
150	7.065	7.142	-289	0.5414	0.7533	75.33
200	7.295	7.329	-357	0.4520	0.7941	79.41
250	8.338	7.764	-281	0.3928	0.8210	82.10
300	8.743	7.822	-284	0.3296	0.8498	84.98

3.4. Linear Polarization Resistance studies

The linear polarization resistance (R_p) values are obtained from the slop of polarization curves. The results show that R_p values are increasing with increase in the concentration of investigated inhibitor and the efficiency values are given in **Table 4**. The inhibition efficiency (IE %) and surface coverage (θ) are calculated in the already mentioned equation. From this study, the highest inhibition efficiency 81.25 % is obtained at the optimum concentration of QSC. The increase in the inhibition efficiencies for corrosion of mild steel in acid solution with increasing concentration can be explained as based on inhibitor adsorption. This study clearly confirmed the formation of a surface film on the working electrode (24).

Table 4: Linear polarization resistance parameters values for corrosion of mild steel in 1 M H₂SO₄ solution containing various concentrations of QSC

Concentration of the Inhibitor (ppm)	R_p (Ωcm^2)	Surface coverage (θ)	IE (%) η
Blank	15	-	-
50	39	0.6154	61.54
100	47	0.6809	68.09
150	57	0.7368	73.68
200	66	0.7727	77.27
250	69	0.7826	78.26
300	80	0.8125	81.25

3.5. Adsorption isotherm studies

The interaction between an inhibitor and the mild steel surface can be described by the adsorption isotherm. To obtain the adsorption isotherm, the degree of surface coverage is calculated from the equation $\theta = \% \text{IE}/100$. The most frequently used adsorption isotherms are Temkin and Langmuir isotherms. The attempts to fit data is obtained from the weight loss studies, EIS, Tafel and LPR studies into different (Temkin and Langmuir) adsorption isotherms revealed that the data best fitted to the Langmuir adsorption isotherm. Many researchers have explained the Langmuir adsorption isotherm with an interaction of adsorbed species on the metallic surfaces (25, 26). According to Langmuir adsorption isotherm, θ is related to C_{inh} by the following equation:

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

where, C_{inh} is the QSC concentration (ppm), θ is the degree of surface coverage and K_{ads} is the equilibrium constant of the adsorption process.

The K_{ads} values can be calculated from the intercept lines on the C_{inh}/θ axis that is related to ΔG_{ads}^0 with the following equation:

$$\Delta G_{ads}^0 = -RT \ln (55.5 K_{ads}) \quad (11)$$

where, R is the universal gas constant ($8.314 \text{ JK}^{-1} \text{ mol}^{-1}$), T is the absolute temperature (K) and 55.5 is the molar concentration of water in the solution in mol L^{-1} (27). The calculated values of ΔG_{ads}^0 , K_{ads} and R^2 are given in **Table 5**. Adsorption of an organic inhibitor on the surface of mild steel can be classified into two main types based on ΔG_{ads}^0 values. If the $\Delta G_{ads}^0 = -20 \text{ kJ mol}^{-1}$ or less value that the adsorption is physical adsorption or physisorption while those around -40 kJ mol^{-1} or more that is chemical adsorption or chemisorption (Karthik et al., 2015). In the present study, the ΔG_{ads}^0 values obtained from all methods are less than -20 kJ mol^{-1} . Therefore, the investigated inhibitor is strongly followed physical adsorption and the process is always spontaneous.

The Langmuir adsorption isotherm was drawn by plotting C_{inh}/θ versus C_{inh} for various concentrations of inhibitor and considering the θ values from WL method, EIS, Tafel polarization and LPR studies at room temperature for mild steel in 1 M H_2SO_4 and the graph is shown in **Figure 7**. The straight line obtained in the graph clearly shows that the investigated inhibitor obeys Langmuir adsorption isotherm.

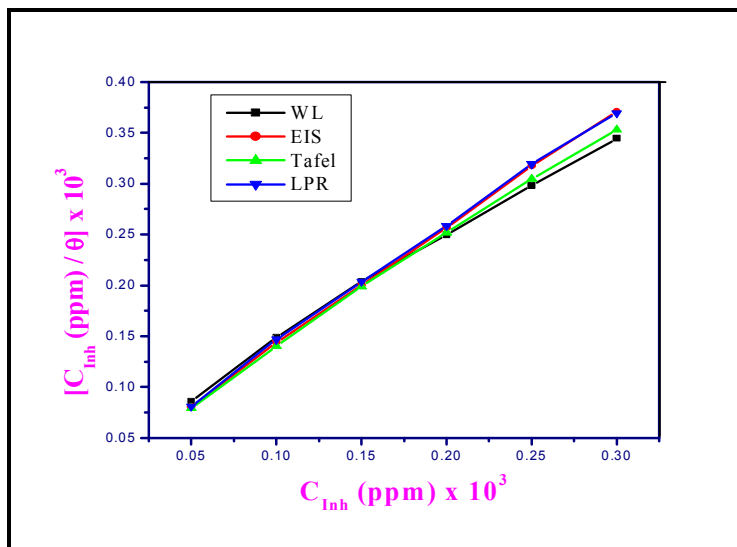


Figure 7: Langmuir plot (using WL, EIS, Tafel and LPR results) for the adsorption of the inhibitor in 1 M H_2SO_4 solution

Table 5: Thermodynamic parameters values derived from Langmuir isotherm for adsorption of optimum concentration of inhibitor on mild steel in 1 M H_2SO_4 solution at room temperature

Method	R^2	$K_{ads} (10^4 \text{ M}^{-1})$	$\Delta G_{ads}^0 (\text{kJ mol}^{-1})$
Weight loss	0.9964	23.5743	-18.08
EIS	0.9995	45.4586	-19.73
Tafel	0.9982	36.6822	-19.19
LPR	0.9988	40.2418	-19.43

3.6. SEM studies

The investigated SEM images of uninhibited and inhibited metal/alloys surface is one of the supportive evidence in adsorption process. The SEM images of mild steel surfaces are shown in **Figure 8 (a-c)**. **Figure 8a** shows the SEM micrograph of polished mild steel in the absence of aggressive media and inhibitor. It shows some very fine scratches, which is obtained during the grinding procedure of mild steel (28, 29). In **Figure 8b** contains number of cracks and pits, because the metallic material is affected by the corrosive environment. However, the presence of optimum concentration of inhibitor in the aggressive medium there are no pits and cracks are observed on the surface of mild steel (**Figure 8c**) that is one of the protective layer is formed between the corrosive sites of the metal and the studied inhibitor. This study confirms that the mild steel surface is

protected from corrosion in the presence of optimum concentration of QSC, which is acting as a good corrosion inhibitor. These SEM results are in good agreement with other studies such as weight loss, electrochemical impedance and polarization measurements.

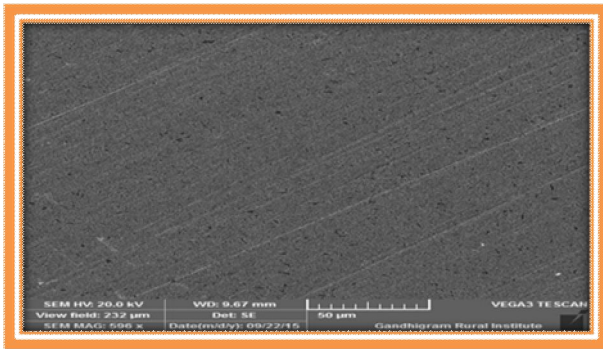
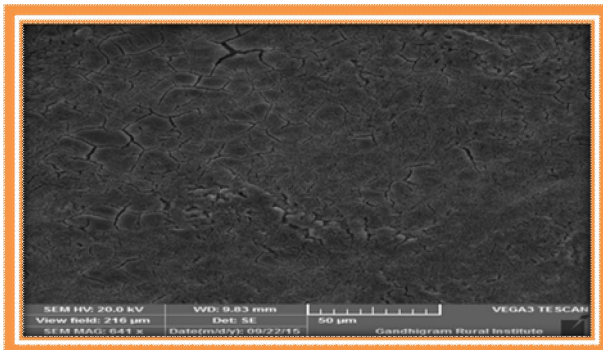
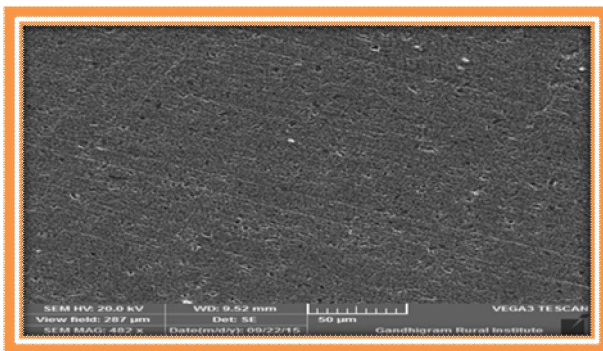
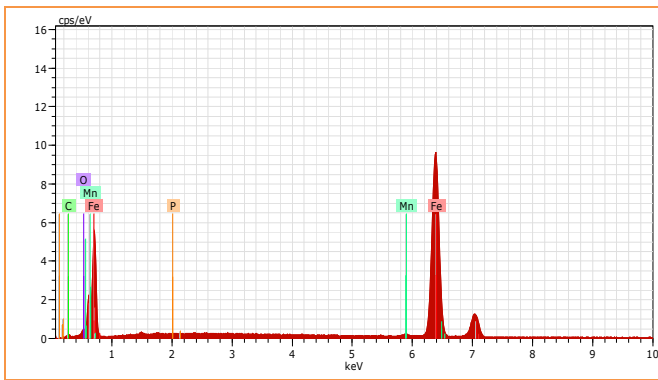
**8a****8b****8c**

Figure 8: SEM surface of mild steel **8a:** Polished mild steel, **8b:** Corroded mild steel and **8c:** Inhibited mild steel

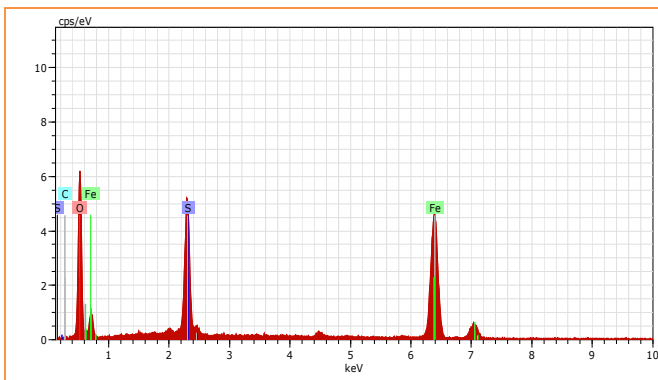
3.7. EDX studies

The formation of atomic peaks on the surface of mild steel is analyzed by using an energy dispersive X-ray spectroscopy (EDX) technique as shown in **Figure 9 (a-c)**. In **Figure 9a**, shows the characteristic peaks of the elements constituting on the surface of polished mild steel without corrosive media and inhibitor. In the absence of inhibitor, **Figure 9b** (corroded mild steel by corrosive media - 1 M H₂SO₄) exhibits the characteristic peaks which are related to Fe, C, Cl and oxygen elements. This additional strong oxygen peak indicated that the formation of corrosion product (metal oxide) on the surface of mild steel. However, the EDX spectra in the presence of optimum concentration of inhibitor, **Figure 9c** (inhibited mild steel) shows some new additional peaks such as nitrogen and sulphur due to the adsorbed layer of QSC that covered the surface of mild steel. In addition, the Fe peaks are suppressed considerably relative to uninhibited (corroded) mild steel specimen. These

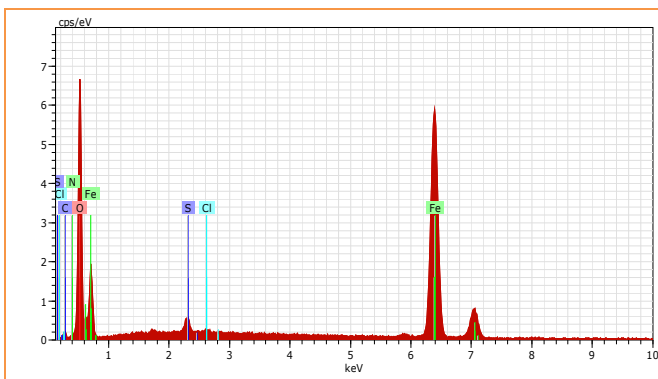
results proved that the adsorption of inhibitor molecule on mild steel surface leads to a decrease of metal oxide layer and control the corrosion process.



9a



9b



9c

Figure 9: EDX spectra of mild steel 9a: Polished mild steel, 9b: Corroded mild steel and 9c: Inhibited mild steel

4. Conclusion

From the above investigation, the following points are suggested,

- The analyzed inhibitor QSC is an effective corrosion inhibitor for mild steel in 1 M H₂SO₄ solution.
- The corrosion inhibition effect is increased with increase in the inhibitor concentration.
- EIS study proved that the investigated inhibitor follows adsorption mechanism and control the rate of corrosion.

- A potentiodynamic polarization study reveals that the studied inhibitor act as mixed-type with slightly cathodic in nature.
- The adsorption of studied inhibitor obeys Langmuir adsorption isotherm. The maximum adsorption found at 300 ppm of QSC in all studies and the maximum efficiency 87.11% is observed in weight loss studies.
- The values of ΔG^0 in all methods indicate that the adsorption of inhibitor on the surface of mild steel is strongly physisorption and the process is spontaneous.
- Inhibition efficiency obtained from weight loss measurements is in good agreement with EIS, Tafel and LPR methods.
- SEM and EDX studies are confirmed the formation of protective layer on the surface of mild steel.

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