**Research Article**

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# Corrosion inhibition of mild steel surface by isoxazoles in HCl solution: Electrochemical studies

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ABSTRACT

The corrosion inhibition efficiency of (3-(4-chlorophenyl isoxazole-5-yl) methanol (CPIM) and (3-(2,4 dichlorophenolisoxazole-5-yl) methanol (DCPIM) for mild steel in 1 M HCl has been studied using electrochemical methods at 40 and 50 °C. Polarization studies showed that the inhibitors are efficient mixed-type corrosion inhibitors, and their inhibition performance increased with the rise of inhibitor concentration and decrease of temperature. The result of EIS measurements was analyzed through an appropriate equivalent circuit model to model the corrosion inhibition.

## 1. Introduction

Inhibitors are promising materials to delay the corrosion process and keep its rate to a minimum amount. In order to reduce the corrosion rate, small quantities of inhibitors were used against corrosion in many environments for various metals like steel [1, 2], iron [3], copper [4], aluminum [5], and zinc [6]. The development of low price, low toxicity, and easy-to-make inhibitors are one of the most demanded methods for protecting metal surfaces against corrosion particularly in industrial centers [7-10]. The chemicals that can act as corrosion inhibitors may be inorganic or organic. Organic molecules are introduced to be effective corrosion inhibitors for many metals and alloys surfaces [11,12]. The experimental studies reported from several literatures have pointed out that the greatest of the effective organic inhibitors used contain nitrogen, oxygen, and sulfur-like hetero-atoms and unsaturated bonds in their structures through which they are formed a protective layer of adsorbed species on the metal surface [13-15]. Coordinate covalent bonds are easily formed between their electron pair and/or π electron cloud and the low-lying vacant d-orbital of metals. The efficiency of the inhibitor molecule on metallic corrosion was dependent on the strength of the newly formed bonds [8,16,17]. The interest in the Schiff-base compounds has grown over the last few years, due to the role of imine bond in their adsorption ability and corrosion inhibition efficiency. Schiff-bases were formed via condensation of an amine with a ketone or aldehyde, which having general formula R-C=N-R′ where R and R′ are aryl, alkyl, or cycloalkyl or heterocyclic groups. Furthermore, the Schiff-bases had a larger inhibition efficiency than the corresponding amines and aldehyde because of both heteroatoms and π electrons have been combined within the same molecule [18,19]. Schiff-bases inhibitors have been previously reported as effective corrosion inhibitors on steel in acid media, such as Schiff-bases such asbenzylidene-pyridine-2-yl-amine [14], 2,2′-[bis-N(4-choloro benzaldimin)]-1,1′-dithio (BCBD) [18], 5-((E)-4-phenylbuta-1,3-dienylideneamino)-1,3,4-thiadiazole-2-thiol (PDTT) [20], furoin thiosemicarbazone (FTSC) [21], 2-(2 hydroxyphenyl)-2,5-diaza-4,6-dimethyl-8-hydroxy-1,5,7-nonatriene [22], (NE)-4-phenoxy-N-(3-phenylallylidene) aniline (PAC) [23], 3-((phenylimino) methyl) quinoline-2-thiol (PMQ) [7], chitosan thiophene carboxaldehyde (ChTSB) [24], 13-bis-[(2-

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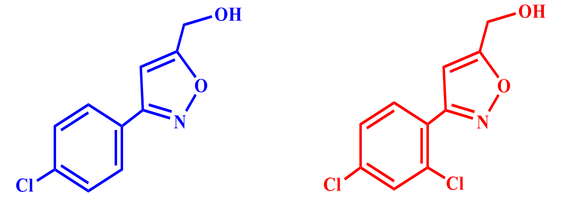
hydroxynaphtaldehyde) 4, 7, 10-trioxatridecanediimine] (HNTTD) [25], 4-((2,3-dichlorobenzylidene) amino)-3-methyl-1H-1, 2, 4-triazole-5(4H)-thione (CBAT) [26], and (4-((thiophene-2-ylmethylene)amino) benzamide) (4-TAB) [27].

Some reported methods suffer from disadvantages. Therefore, to avoid these limitations, the finding of easily available compounds with high corrosion inhibition activity is still desirable. Because of these reasons and also as a part of our ongoing research program on the application of various chemical compounds for the development of useful synthetic corrosion inhibitors [28-30], herein, we report the inhibitory behavior of two isoxazoles that were synthesized and investigated at 30 °C in our previous paper [31] from 4-chlorobenzaldoxime or 2,4-chlorobenzaldoxime and propargyl alcohol, named (3-(4-chlorophenyl isoxazole-5-yl) methanol (CPIM) and (3-(2,4 dichlorophenolisoxazole-5-yl) methanol (DCPIM), respectively (Scheme 1).

The research was done on mild steel in 1.0 M HCl solution and at different temperatures by Tafel polarization data and electrochemical impedance spectroscopy (EIS).Compared to our previous work, the effect of increasing temperature was studied by carrying out the corrosion inhibition process at different temperatures (40 and 50 °C).



(3-(4-Chlorophenyl isoxazole-5-yl) methanol (CPIM)

(3-(2,4 Dichlorophenolisoxazole-5-yl) methanol (DCPIM)

**Scheme 1.** The chemical structure of the investigated compounds

## 2. Experimental:

### 2.1. Materials:

### 2.1.1. Mild steel substrates:

Mild steel samples obtained from the commercial MS bars were used as the working electrodes for potentiodynamic polarization and EIS methods throughout this study. MS samples with a size of 1cm×1cm×1cm were coated with polyester resin, having an exposed area of 1 $cm^2$.

### 2.1.2. Synthesis of the isoxazoles as inhibitors:

The isoxazoles, namely (3-(4-chlorophenyl isoxazole-5-yl) methanol and (3-(2,4 dichlorophenolisoxazole-5-yl) methanol was prepared by reacting 4-chlorobenzaldoxime or 2,4-chlorobenzaldoxime and propargyl alcohol as previously described [31,32]. A solution of 4-chlorobenzaldoxime or 2,4-chlorobenzaldoxime in anhydrous dichloromethane was mixed with a solution of N-chlorosuccinimide in dichloromethane. The resultant solution was left to be stirred for 1h at 25 °C. Then, a mixture of propargyl alcohol and trimethylamine was added drop wise to the obtained liquid with mechanical stirring at room temperature. The mixture was refluxed for 6 h to complete the reaction. After completion of the reaction, the mixture was cooled to room temperature and washed with pure water and dried with sodium carbonate.

### 2.2. Methods:

### 2.2.1. Electrochemical measurements:

The potentiodynamic polarization and EIS measurements were recorded by using an AUTOLAB potentiostat-galvanostat/Nova software. All electrochemical tests were performed at room temperature under environmental pressure utilizing 100 mL of corrosive solution. Tafel polarization and EIS measurements were performed in a double-wall one compartment cell with a three-electrode configuration; a mild steel working electrode with a 1$cm^2$ surface area, a platinum auxiliary electrode as a counter electrode (CE), and an Ag/AgCl saturated calomel electrode (SCE) as a reference electrode. The potential scan rate was 2 $mVs^{-1}$.The potential was scanned in the maximum overvoltage of 200 mV. Impedance data were analyzed, and the alternating current frequency range was extended from 1 to 10MHz, with a 10 mV sine wave AC voltage as the excitation signal. The effect of temperature and concentration of isoxazole on the inhibition performance for mild steel in 1 M HCl at temperatures 40 and 50 °C was studied using impedance and Tafel polarization measurements.

## 3. Results and discussion:

### 3.1. Polarization measurement:

The polarization profiles of mild steel in 1.0 M HCl solution at 40 and 50 °C for various CPIM and DCPIM concentrations are shown in Fig. 1, 2, 3, and 4, respectively. Polarization curves were showed as the plots of potential against the logarithm of current density. The corrosion parameters such as corrosion current density ($I_{corr}$), corrosion potential ($E_{corr}$), anodic and cathodic Tafel slopes ($\beta_a$ and $\beta_c$), polarization resistance (Rp), and percentage inhibition efficiency (IE%) are listed in Table 1 and 2. The percentage inhibition efficiency (*IE*%) was calculated using the following equation 1:

$$IE\% = \left[1 - \left(\frac{I_{corr}}{I°_{corr}}\right)\right] \times 100 = \frac{R_p - R°_p}{R_p} \times 100 \quad (1)$$

Where $I°_{corr}$ and $I_{corr}$ are the corrosion current density values estimated by the intersection of the extrapolated

Tafel lines and the corrosion potential without and with the corrosion inhibitor in acid solution, respectively. $R_p°$ and $R_p$ are the polarization resistance in the absence and presence of an inhibitor molecule. The $R_P$ values were determined from the $\beta_a$ and $\beta_c$ for the anodic and cathodic branches by linear polarization resistance (LPR) technique displayed in the following equation 2:

$$R_p = \frac{\beta_a \beta_c}{2.303(\beta_a + \beta_c)} \times \frac{1}{I_{corr}} \qquad (2)$$

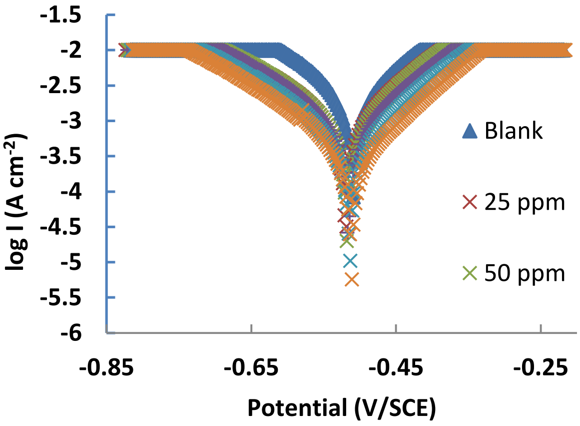


**Figure1.** Tafel polarization plots for mild steel in 1 M HCl solution in the absence (blank) and the presence of different concentrations of CPIM at the temperature of 40 °C.

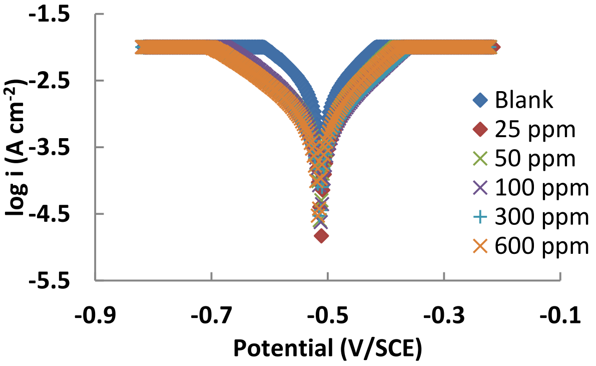


**Figure 2.** Tafel polarization plots for mild steel in 1 M HCl solution in the absence (blank) and the presence of different concentrations of DCPIM at the temperature of 40 °C.

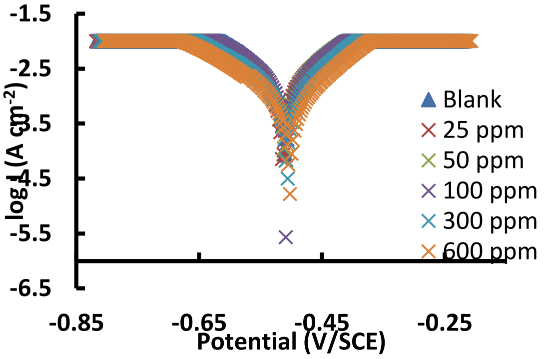


**Figure 3.** Tafel polarization plots for mild steel in 1 M HCl solution in the absence (blank) and the presence of different concentrations of CPIM at the temperature of 50 °C.

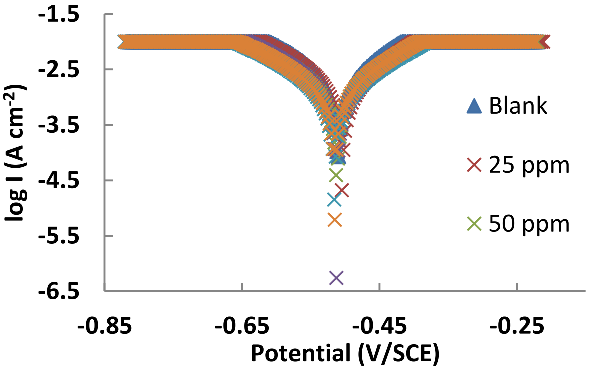


**Figure 4.** Tafel polarization plots for mild steel in 1 M HCl solution in the absence (blank) and the presence of different concentrations of DCPIM at the temperature of 50 °C.

It is clear that $I_{corr}$ for anodic and cathodic reactions decreased with increasing the concentration of the CPIM and DCPIM. This effect is ascribed to the inhibitor adsorption over the active sites of the mild steel surface, and surface area available for $H^+$ ions decreases [33,34]. The heteroatoms and unsaturated bonds in the isoxazoles form this protective layer on the mild steel surface. An inhibitor can be classified as anodic, cathodic, or mixed type inhibitor based on their type of reaction usually occurs in acidic solution. The anodic reaction is the metal dissolution, and cathodic reaction is the proton-discharge reaction or release of hydrogen gas [34]. The addition of inhibitor affects either the anodic or cathodic reaction, or sometimes both. When the difference between the $E_{corr}$ value differences between the mild steel electrode with

**Table 1**. Corrosion parameters for the mild steel at different concentrations of the CPIM in 1M HCl solution in temperature of 40 °C by potentiodynamic polarization method.

| *Concentration* | *$R_p$* | *Ba* | *βc* | *$I_{corr}$* | *-$E_{corr}$* | *IE%* |
|---|---|---|---|---|---|---|
| **Blank** | 22.68 | 57.28 | 70.48 | 560.59 | 513.46 | - |
| **25 ppm** | 38.69 | 77.33 | 92.18 | 472.05 | 518.59 | 41.38 |
| **50 ppm** | 42.73 | 57.76 | 59.06 | 377.99 | 517.91 | 48.13 |
| **100 ppm** | 45.65 | 74.65 | 84.93 | 290.02 | 514.05 | 50.13 |
| **300 ppm** | 76.39 | 83.95 | 91.63 | 249.08 | 512.60 | 70.31 |
| **600 ppm** | 96.71 | 101.64 | 116.69 | 243.92 | 505.94 | 76.55 |

**Table 2**. Corrosion parameters for the mild steel at different concentrations of the DCPIM in 1M HCl solution in temperature of 40 °C by potentiodynamic polarization method.

| *Concentration* | $R_p$ | *βa* | *βc* | $I_{corr}$ | $-E_{corr}$ | *IE%* |
|---|---|---|---|---|---|---|
| **Blank** | 22.68 | 57.28 | 70.48 | 560.59 | 513.46 | - |
| **25 ppm** | 44.14 | 60.68 | 69.40 | 506.68 | 513.27 | 48.62 |
| **50 ppm** | 49.26 | 101.79 | 132.02 | 318.51 | 509.92 | 53.96 |
| **100 ppm** | 51.32 | 46.56 | 52.49 | 265.26 | 507.55 | 55.81 |
| **300 ppm** | 56.26 | 67.71 | 69.77 | 208.78 | 506.66 | 59.68 |
| **600 ppm** | 46.35 | 70.29 | 87.75 | 623.43 | 505.35 | 51.25 |

**Table 3**. Corrosion parameters for the mild steel at different concentrations of the CPIM in 1M HCl solution in temperature of 50 °C by potentiodynamic polarization method.

| *Concentration* | $R_p$ | *βa* | *βc* | $I_{corr}$ | $-E_{corr}$ | *IE%* |
|---|---|---|---|---|---|---|
| **Blank** | 16.77 | 62.27 | 55.23 | 757.95 | 531.68 | - |
| **25 ppm** | 20.13 | 74.63 | 80.21 | 834.03 | 513.60 | 16.68 |
| **50 ppm** | 21.03 | 71.30 | 73.24 | 745.88 | 510.55 | 20.27 |
| **100 ppm** | 21.26 | 52.19 | 44.77 | 492.16 | 509.99 | 21.13 |
| **300 ppm** | 32.35 | 63.93 | 73.59 | 459.27 | 505.21 | 48.15 |
| **600 ppm** | 42.02 | 77.30 | 94.36 | 439.10 | 499.87 | 60.09 |

**Table 4**. Corrosion parameters for the mild steel at different concentrations of the DCPIM in 1M HCl solution in temperature of 50 °C by potentiodynamic polarization method.

| *Concentration* | $R_p$ | *βa* | *βc* | $I_{corr}$ | $-E_{corr}$ | *IE%* |
|---|---|---|---|---|---|---|
| **Blank** | 16.77 | 62.27 | 55.23 | 757.95 | 531.68 | - |
| **25 ppm** | 24.19 | 60.73 | 59.23 | 601.31 | 524.60 | 30.66 |
| **50 ppm** | 27.64 | 78.12 | 75.04 | 538.37 | 513.63 | 39.32 |
| **100 ppm** | 30.27 | 57.46 | 49.07 | 379.75 | 513.23 | 44.60 |
| **300 ppm** | 34.54 | 54.74 | 49.20 | 325.75 | 505.34 | 51.46 |
| **600 ppm** | 30.32 | 51.64 | 50.30 | 394.91 | 506.85 | 44.69 |

and without protective film is at least 85 mV, the inhibitor molecules are acted as mixed type inhibitor [16,35]. Herein, the addition of CPIM and DCPIM shifted the $E_{corr}$, with respect to the blank, but this shift is about -35 mV and -26 mV, respectively. The results of Tables 1-4 clearly demonstrate that βa and βc values did not show significant changes. This result indicated that the anodic and cathodic corrosion reaction mechanism did not change [36]. Also, with increasing the concentration of all the two inhibitor molecules, the inhibition efficiency increased for mild steel at studied temperatures. The high *IE%* value suggested a full coverage of the studied steel surface with different doses of inhibitors CPIM and DCPIM at 40 and 50 °C. The influence of the reaction temperature on the potentiodynamic polarization profiles in the absence and presence of different concentrations of CPIM and DCPIM for mild steel in 1 M HCl is comparable in Tables 1-4. It is quite cleared that the corrosion current densities increased with the increase of temperature in 1 M HCl solution without and with 25 ppm of the investigated inhibitors. As a result, the inhibition efficiency and surface coverage degree decreased with the rise of temperature.

## 3.2. Electrochemical impedance spectroscopy (EIS):

The corrosion of mild steel surface with and without inhibitors between the concentration range 25-600 ppm in 1 M HCl solution is investigated by electrochemical impedance spectroscopy at 40 and 50 °C. Nyquist plots of mild steel in the absence and presence of CPIM and DCPIM at 40 °C are given in Fig. 5 and 6. The Nyquist plot of mild steel obtained in a blank solution was added in Fig. 5 and 6 as an inset. The Nyquist diagrams show a little depressed semicircular shape at all frequencies range, which indicates the formation of a barrier on the surface and the corrosion of mild steel is mainly controlled by the electron transfer process [37].

A similar corrosion inhibition mechanism has been reported in the literature for the corrosion of mild steel in 1 M HCl in the presence of studied inhibitors [23, 38].

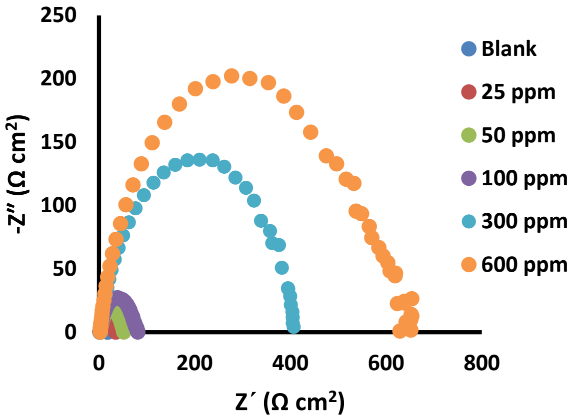


**Figure 5.** Nyquist plots of mild steel in 1.0 MHCl solutions without and with various concentrations of CPIM at 40 °C.

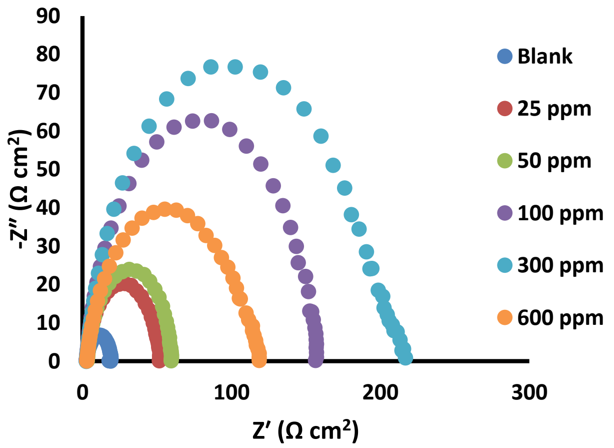


**Figure 6.** Nyquist plots of mild steel in 1.0 M HCl solutions without and with various concentrations of DCPIM at 40 °C.

The diameter of semicircle increases significantly when the isoxazoles are added to the solution. The diameter of semicircle is parallel increased in the presence of the inhibitors. This phenomenon proposes the creation of a protective layer on the mild steel surface. An electrical equivalent circuit was presented to explain the EIS data, as shown in Fig. 7.

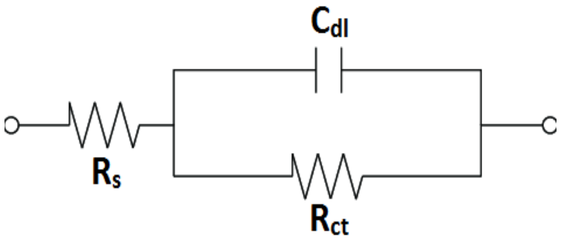


**Figure 7.** The proposed equivalent circuit model for the investigated system.

This circuit is generally used for modeling of the iron/acid interface model. In this circuit $R_s$ solution resistance, $R_{ct}$ charge transfer resistance, and $C_{dl}$ is double-layer capacitance. This type of Nyquist diagram was considered as distributed capacitance. To obtain a non-ideal behavior and representative fit, the $C_{dl}$ was replaced by the constant phase element (*CPE*) [23,39]. The impedance function of the CPE can be expressed as follows:

$$Z_{CPE} = [(Y_o(jw)_n]^{-1} \qquad (3)$$

Where $Y_o$ is a proportionality factor, *w* is an angular frequency, $j^2 = -1$ is an imaginary number, and n is the phase shift. In the Nyquist plot, the difference in the lower frequency and higher frequency in the real impedance is generally considered as $R_{ct}$. The inhibition efficiency gained by the EIS measurements was computed by the following relation:

**Table 5**. Electrochemical parameters obtained from measurements of EIS for mild steel corrosion in 1 M HCl solution in various concentrations of CPIM inhibitor at 40 °C.

| *Concentration* | $R_s$ *(Ω/cm²)* | $CPE_{(dl)}$ *(μF/cm²)* | *n* | $R_p$ *(Ω/cm²)* | *IE%* |
|---|---|---|---|---|---|
| **Blank** | 2.55 | 0.000235 | 0.83 | 17.10 | - |
| **25 ppm** | 2.65 | $8.05\times10^{-5}$ | 0.86 | 32.08 | 46.69 |
| **50 ppm** | 2.59 | $8.61\times10^{-5}$ | 0.84 | 47.29 | 63.84 |
| **100 ppm** | 2.46 | $7.91\times10^{-5}$ | 0.84 | 74.57 | 76.69 |
| **300 ppm** | 2.87 | $5.40\times10^{-5}$ | 0.82 | 387.3 | 95.58 |
| **600 ppm** | 2.73 | $4.04\times10^{-5}$ | 0.84 | 550.7 | 96.89 |

**Table 6**. Electrochemical parameters obtained from measurements of EIS for mild steel corrosion in 1 M HCl solution in various concentrations of DCPIM inhibitor at 40 °C.

| *Concentration* | $R_s$ *(Ω/cm²)* | $CPE_{(dl)}$ *(μF/cm²)* | *n* | $R_p$ *(Ω/cm²)* | *IE%* |
|---|---|---|---|---|---|
| **Blank** | 2.55 | 0.000235 | 0.83 | 17.10 | - |
| **25 ppm** | 2.68 | $6.71\times10^{-5}$ | 0.88 | 48.80 | 64.95 |
| **50 ppm** | 2.62 | $6.20\times10^{-5}$ | 0.89 | 56.82 | 69.90 |

| | | | | | |
|---|---|---|---|---|---|
| **100 ppm** | 2.67 | $7.35\times10^{-5}$ | 0.88 | 153.3 | 88.84 |
| **300 ppm** | 3.28 | $6.66\times10^{-5}$ | 0.87 | 196.3 | 91.28 |
| **600 ppm** | 2.79 | $9.04\times10^{-5}$ | 0.82 | 110.1 | 84.46 |

**Table 7**. Electrochemical parameters obtained from measurements of EIS for mild steel corrosion in 1 M HCl solution in various concentrations of CPIM inhibitor at 50 °C.

| ***Concentration*** | $R_s$ $(\Omega/cm^2)$ | $CPE_{(dl)}$ $(\mu F/cm^2)$ | $n$ | $R_p$ $(\Omega/cm^2)$ | *IE%* |
|---|---|---|---|---|---|
| **Blank** | 2.79 | 0.000280 | 0.82 | 14.49 | - |
| **25 ppm** | 3.08 | $8.22\times10^{-5}$ | 0.87 | 27.14 | 46.61 |
| **50 ppm** | 3.24 | $8.02\times10^{-5}$ | 0.87 | 28.33 | 48.83 |
| **100 ppm** | 3.18 | 0.000109 | 0.87 | 56.32 | 74.27 |
| **300 ppm** | 3.64 | $8.01\times10^{-5}$ | 0.87 | 80.35 | 81.96 |
| **600 ppm** | 3.31 | 0.000146 | 0.81 | 54.35 | 73.33 |

**Table 8**. Electrochemical parameters obtained from measurements of EIS for mild steel corrosion in 1 M HCl solution in various concentrations of DCPIM inhibitor at 50 °C.

| ***Concentration*** | $R_s$ $(\Omega/cm^2)$ | $CPE_{(dl)}$ $(\mu F/cm^2)$ | $n$ | $R_p$ $(\Omega/cm^2)$ | *IE%* |
|---|---|---|---|---|---|
| **Blank** | 2.45 | 0.000280 | 0.82 | 14.49 | - |
| **25 ppm** | 2.16 | 0.000135 | 0.87 | 27.14 | 46.61 |
| **50 ppm** | 2.22 | 0.000147 | 0.87 | 28.33 | 48.83 |
| **100 ppm** | 2.23 | 0.000120 | 0.87 | 56.32 | 74.27 |
| **300 ppm** | 2.37 | $6.86\times10^{-5}$ | 0.87 | 80.35 | 81.96 |
| **600 ppm** | 2.29 | $5.02\times10^{-5}$ | 0.81 | 54.35 | 73.33 |

**Table 1**. Corrosion parameters for the mild steel at different concentrations of the CPIM in 1M HCl solution in

$$IE\% = \left[\left(1 - \frac{R_{ct}}{R^{\circ}_{ct}}\right)\right] \times 100 \qquad (4)$$

Where $R_{ct}$ and $R^{\circ}_{ct}$ are the charge transfer resistances in uninhibited and inhibited solution, respectively. In the other investigated temperature, Nyquist plots of mild steel in the absence and presence of CPIM and DCPIM at 50 °C are given in Figs. 8 and 9.

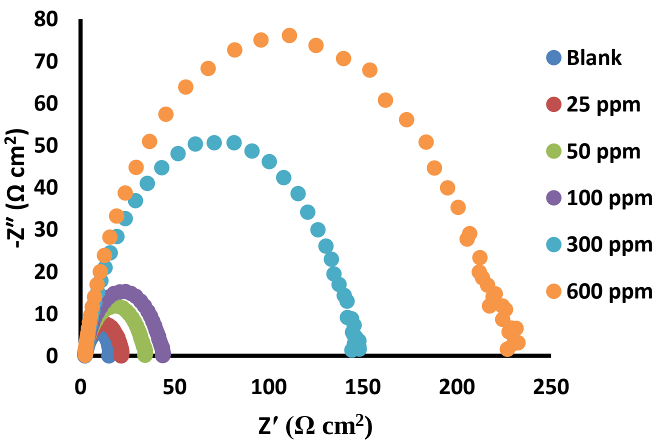


**Figure 8.** Nyquist plots of mild steel in 1.0 M HCl solutions without and with various concentrations of CPIM at 50 °C.

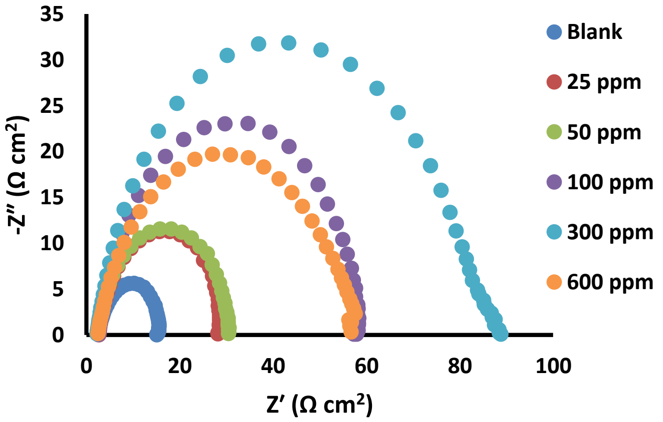


**Figure 9.** Nyquist plots of mild steel in 1.0 MHCl solutions without and with various concentrations of DCPIM at 50 °C.

Tables 5 and 6 lists the impedance parameters of the Nyquist plots of the CPIM and DCPIM in different concentrations at 40 °C. The *Rp* values for mild steel are observed to increase with additive in 1.0 M HCl solution that may be due to adsorption of the CPIM and DCPIM molecules from the solution on the electrode surface. The transmission arises in a large amount with increasing inhibitor concentration in bulk solution. This layer creates a barrier for charge transfer at the metal-solution interface. The decrease in $C_{dl}$ values with the increase in inhibitor concentrations is due to the increase in the thickness of the electric double-layer. The values of inhibition efficiency (*IE%*) at 40 °C increase with the concentration of these isoxazole inhibitors up to 96.89 % for CPIM, and 91.28% for DCPIM, when the concentration reaches 600 and 300 ppm, respectively.

## Conclusion:

The CPIM and DCPIM isoxazoles were synthesized and investigated as a corrosion inhibitor for mild steel in 1 MHCl solution using a series of techniques like potentiodynamic polarization and electrochemical impedance spectroscopy (EIS). CPIM and DCPIM have a good inhibition effect for corrosion of mild steel in acidic solutions to form a protective film. The inhibition efficiency increases with increasing inhibitor concentration. The potentiodynamic polarization curves indicated that all studied isoxazoles behaved as a mixed type corrosion inhibitor by inhibiting both anodic metal dissolution and cathodic hydrogen evolution reactions. Corrosion inhibition efficiencies are in the order of(CPIM>DCPIM) at 40 and 50 °C. The results indicated that the only para-substituted compound had the highest inhibition efficiency but was reduced proportionally with temperature. The electrochemical impedance study presented that corrosion inhibition of mild steel takes place by the adsorption process.

## Acknowledgments

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## Conflicts of Interest

The authors declare no conflict of interest.

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