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Research Article

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3,7-Dimethylquinoxalin-2-(1*H*)-one for inhibition of acid corrosion of carbon steel

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ABSTRACT

Electrochemical and impedance experiments were carried out to evaluate the corrosion behavior of carbon steel in aerated 1.0 M HCl solutions in the presence of 3,7-dimethylquinoxalin-2-(1H)-one (DQO) at 308K. The corrosion rate of carbon steel was found to increase in the presence this compound. The adsorption and inhibitory effect of DQO on carbon steel surface in aerated 1.0 M HCl solutions were then investigated in detail by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS). The data obtained indicate that quinoxaline behaves as a mixed-type inhibitor adsorbing onto the carbon steel surface according to the Langmuir isotherm, with the negative ΔG°_{ads} *value of -47.47 kJ mol*$^{-1}$ *signifying a spontaneous adsorption process. The corrosion inhibition efficiency increased with DQO concentration in the range of 10*$^{-6}$*-10*$^{-3}$ *M.*

Keywords: Carbon steel; Corrosion; HCl; Quinoxaline; EIS; Potentiodynamic polarization.

INTRODUCTION

Many organic molecules are recognized as corrosion inhibitors for metals and alloys. It is well-known that their effectiveness is closely related to their adsorption. The adsorption of organic compounds onto the surface of corroding metals markedly modifies the properties of the metal/solution interface, such as the double layer capacitance and the kinetics of the interfacial electron transfer process [1]. As a consequence, the relationship between the adsorption and corrosion inhibition is important in surface science studies. In addition, new corrosion inhibitors for the protection of metals and alloys in different media are continually being sought due to their importance and versatility. They also facilitate the protection of metal parts that have complex shapes with difficult direct access to the whole surface area.

In general, the stronger the affinity between the metallic material and the organic molecule, the larger the active surface area covered by the protective film formed by the inhibitor. The interactions between metals and inhibitors can occur by chemical and/or physical adsorption of the organic compound on the metal surface. The former is facilitated by the presence of electron-acceptor vacant d orbitals in the metal (a Lewis acid) which participate in the formation of coordinated bonds with electron-donor inhibitors (a Lewis base), whereas the latter is strongly dependent on the molecular weight and chemical structure of the molecule. The performance of organic corrosion inhibitors is, therefore, often related to the presence of π electrons and hetero-atoms such as sulfur, oxygen and nitrogen in the molecule [2-22].

The objective of this investigation is to determine the corrosion inhibition efficiency of 3,7-dimethylquinoxalin-2-(1H)-one (DQO) as a novel inhibitor for the corrosion of carbon steel in 1.0 M HCl. The inhibition efficiency was determined using two different techniques: electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization techniques. The chemical structure of the studied Quinoxaline derivative is given in Fig 1.

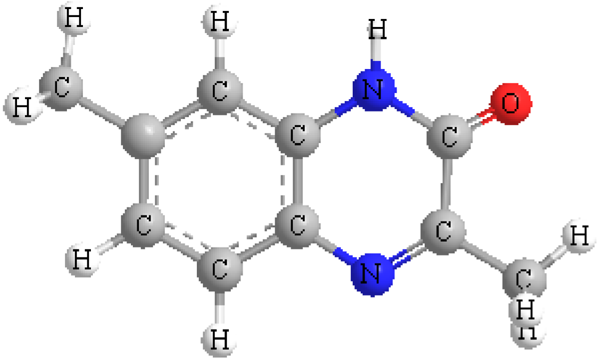

Figure 1. Chemical structure of the studied organic compound.

EXPERIMENTAL SECTION

Materials

The steel used in this study is a carbon steel (CS) (Euronorm: C35E carbon steel and US specification: SAE 1035) with a chemical composition (in wt%) of 0.370 % C, 0.230 % Si, 0.680 % Mn, 0.016 % S, 0.077 % Cr, 0.011 % Ti, 0.059 % Ni, 0.009 % Co, 0.160 % Cu and the remainder iron (Fe).

Solutions

The aggressive solutions of 1.0 M HCl were prepared by dilution of analytical grade 37% HCl with distilled water. The concentration range of 3,7-dimethylquinoxalin-2-(1H)-one (DQO) used was 10^{-6} M to 10^{-3} M.

Polarization measurements

Electrochemical impedance spectroscopy

The electrochemical measurements were carried out using Volta lab (Tacussel- Radiometer PGZ 100) potentiostate and controlled by Tacussel corrosion analysis software model (Voltamaster 4) at under static condition. The corrosion cell used had three electrodes. The reference electrode was a saturated calomel electrode (SCE). A platinum electrode was used as auxiliary electrode of surface area of 1 cm^2. The working electrode was carbon steel. All potentials given in this study were referred to this reference electrode. The working electrode was immersed in test solution for 30 minutes to a establish steady state open circuit potential (*E*ocp). After measuring the *E*ocp, the electrochemical measurements were performed. All electrochemical tests have been performed in aerated solutions at 308 K. The EIS experiments were conducted in the frequency range with high limit of 100 kHz and different low limit

0.1 Hz at open circuit potential, with 10 points per decade, at the rest potential, after 30 min of acid immersion, by applying 10 mV ac voltage peak-to-peak. Nyquist plots were made from these experiments. The best semicircle can be fit through the data points in the Nyquist plot using a non-linear least square fit so as to give the intersections with the *x*-axis.

The inhibition efficiency of the inhibitor was calculated from the charge transfer resistance values using the following equation [23]:

$$\eta_z\% = \frac{R_{ct}^{i} - R_{ct}^{\circ}}{R_{ct}^{i}} \times 100 \qquad (1)$$

where, R_{ct}° and R_{ct}^{i} are the charge transfer resistance in absence and in presence of inhibitor, respectively.

Potentiodynamic polarization

The electrochemical behaviour of carbon steel sample in inhibited and uninhibited solution was studied by recording anodic and cathodic potentiodynamic polarization curves. Measurements were performed in the 1.0 M HCl solution containing different concentrations of the tested inhibitor by changing the electrode potential automatically from -600 to -250 mV versus corrosion potential at a scan rate of 1 mV s^{-1}. The linear Tafel segments of anodic and cathodic curves were extrapolated to corrosion potential to obtain corrosion current densities (I_{corr}). From the polarization curves obtained, the corrosion current (I_{corr}) was calculated by curve fitting using the equation:

$$I = I_{corr}\left[exp\left(\frac{2.3\Delta E}{\beta_a}\right) - exp\left(\frac{2.3\Delta E}{\beta_c}\right)\right] \qquad (2)$$

The inhibition efficiency was evaluated from the measured I_{corr} values using the relationship:

$$\eta_{Tafel}\% = \frac{I^{\circ}_{corr} - I^{i}_{corr}}{I^{\circ}_{corr}} \times 100 \qquad (3)$$

where, I°_{corr} and I^{i}_{corr} are the corrosion current density in absence and presence of inhibitor, respectively.

RESULTS AND DISCUSSION

Potentiodynamic polarization

The kinetics of the anodic and cathodic reactions occurring on carbon steel electrodes in 1.0 M HCl solutions with different 3,7-dimethylquinoxalin-2-(1H)-one concentrations was studied through the polarization measurements. The complete potentiodynamic polarization curves are shown in Fig. 2. The electrochemical parameters, i.e. corrosion current density (I_{corr}), cathodic (β_c) Tafel constants, potential of corrosion (E_{corr}) and inhibition efficiencies (η_{Tafel} (%)), shown in Table 1, were collected from Tafel plots and polarization resistance experiments carried out separately.

Table 1 Polarization data of carbon steel in 1.0 M HCl without and with addition of inhibitor at 308 K.

Inhibitor	Concentration (M)	$-E_{corr}$ (mV/SCE)	$-\beta_c$ (mV dec^{-1})	I_{corr} (μA cm^{-2})	η_{Tafel} (%)
Blank	1.0	475.9	175.6	1077.8	-
DQO	10^{-3}	481.9	165.8	21.8	98.0
	10^{-4}	488.3	163.1	43.7	96.0
	10^{-5}	499.0	162.5	79.0	92.7
	10^{-6}	510.2	170.6	116.3	89.2

From Fig. 2 and Table 1, it's clearly, I_{corr} decreased remarkably while η_{Tafel} increased with increasing of the inhibitor concentration, and the maximum (η_{Tafel}) is up to 98.0 % at 1×10^{-3} M of inhibitor. There is no definite trend in the shift of E_{corr} in the presence of corrosion inhibitor, therefore, the synthesized inhibitor can be arranged as a mixed-type inhibitor, and the inhibition action is caused by geometric blocking effect [24]. Namely, the inhibition action comes from the reduction of the reaction area on the surface of the corroding metal.

Furthermore, in the presence of either compound, the slight change of β_c has indicated that the corrosion mechanism of carbon steel does not change. In other words, this inhibitor has decreased the active surface area for the acid corrosion attack without affecting the mechanism of corrosion and only caused inactivation of a part of the metal surface with respect to the corrosive medium.

In acidic solutions, it is known that inhibitor molecules can be protonated. Thus in solution both neutral molecule and cationic forms of inhibitor exist [25, 26]. In hydrochloric acid solutions, anodic dissolution of iron takes following steps:

(i)

$$Fe + Cl^- \rightleftharpoons (FeCl^-)_{ads}$$

(ii)

$$(FeCl^-)_{ads} \rightleftharpoons (FeCl)_{ads} + e^-$$

(iii)

$$(FeCl)_{ads} \rightarrow (FeCl^{+}) + e^{-}$$

(iv)

$$(FeCl^{+}) \square \quad Fe^{++} + Cl^{-}$$

And cathodic hydrogen evolution takes following steps:

(v)

$$Fe + H^{+} \square \quad (FeH^{+})_{ads}$$

(vi)

$$(FeH^{+})_{ads} + e^{-} \rightarrow (FeH)_{ads}$$

(vii)

$$(FeH)_{ads} + H^{+} + e^{-} \rightarrow Fe + H_{2}$$

It is assumed that Cl^- ion is first adsorbed onto the positively charged metal surface by coulombic attraction and then inhibitor molecules can be absorbed through electrostatic interactions between the positively charged molecules and the negatively charged metal surface [25]. These adsorbed molecules interact with $(FeCl^-)_{ads}$ species to form monomolecular layers (by forming a complex) on the steel surface. These layers protect carbon steel surface from attack by chloride ions. Thus, the oxidation of $(FeCl^-)_{ads}$ into Fe^{++} can be prevented. On the other hand, the protonated inhibitor molecules are also adsorbed at cathodic sites in competition with hydrogen ions that going to reduce hydrogen evolution.

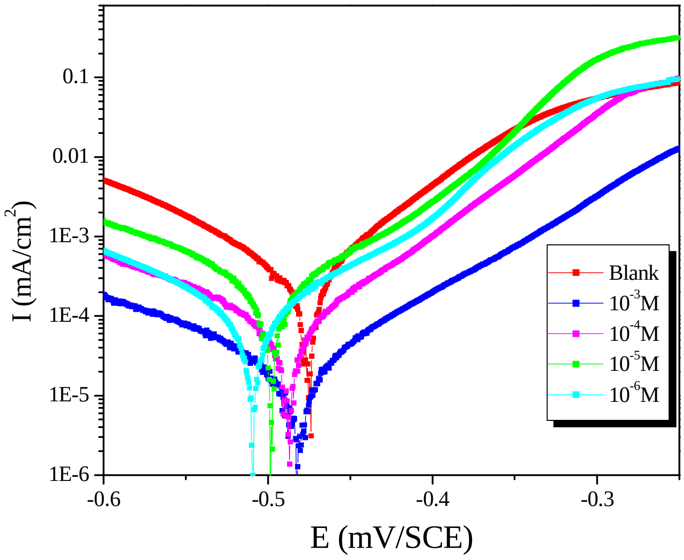

Figure 2. Polarisation curves of carbon steel in 1.0 M HCl for various concentrations of DQO.

Electrochemical impedance spectroscopy measurements

Electrochemical impedance spectroscopy (EIS) is a well-established and powerful tool in the study of corrosion. Surface properties, properties, electrode kinetics and mechanistic information can be obtained from the impedance diagrams [27]. Fig. 3 shows the Nyquist plot obtained at the open-circuit potential, while Table 2 summarizes the impedance data extracted from EIS experiments carried out both in the absence and presence of increasing concentrations of 3,7-dimethylquinoxalin-2-(1H)-one. In 3,7-dimethylquinoxalin-2-(1H)-one -free solution, a depressed charge transfer semicircle is observed at high frequency, which is attributed to the time constant of the charge transfer and double- layer capacitance [27,28]. The intersection of this semicircle with the real axis at high frequencies furnished a value of 1.67 Ω cm^2 for the ohmic resistance (R_s) of the solution enclosed between the working electrode and the tip of the salt bridge containing the reference electrode. At low frequencies, a charge transfer resistance (R_{ct}) of 31.0 Ω cm^2 was found from the difference in impedances at lower and higher frequencies. The double-layer capacitance (C_{dl}) was calculated from the equation below:

$$C_{dl} = \frac{1}{2\pi f_{\max} R_{ct}} \tag{4}$$

where f_{max} is the frequency value at which the imaginary component of the impedance is maximal. A C_{dl} value of 80.99 µF cm^{-2} was found for the carbon steel electrode in the blank 1.0 M HCl solution. The values observed for R_s, R_{ct} and C_{dl} for carbon steel in corrosion inhibitor-free solutions are in agreement with the values reported by other authors for similar conditions [29,30]. The Nyquist plots shown in Fig. 2 for carbon steel electrodes immersed in 1.0 M HCl solutions containing 10^{-3}, 10^{-4}, 10^{-5} and 10^{-6} M of 3,7-dimethylquinoxalin-2-(1H)-one depict a behavior analogous to that observed in solutions without the inhibitor, indicating that mainly a charge transfer process controls the corrosion of carbon steel. Deviations from a perfect circular shape indicate frequency dispersion of interfacial impedance arising from a lack of homogeneity of the electrode surface due to roughness or interfacial phenomena [31]. However, it is worth noting that the impedances increase in the presence of 3,7-dimethylquinoxalin-2-(1H)-one, but the electrochemical reactions responsible for corrosion do not change. These results support those obtained from the Tafel experiments for lower 3,7-dimethylquinoxalin-2-(1H)-one concentration and confirm the inhibitor adsorption onto the carbon steel surface. As can be seen in Table 2, slightly higher R_s values were obtained in the presence of the corrosion inhibitor. This was to be expected because, in general, organic compounds reduce the dielectric constant of aqueous solutions, increasing their resistance. More important, however, are the observations related to C_{dl} and R_{ct}. The EIS results clearly indicate that 3,7-dimethylquinoxalin-2-(1H)-one decreases the double-layer capacitance and increases the charge transfer resistance; as consequence a larger diameter of the semicircle is observed in Nyquist plots. The decrease in C_{dl} can be interpreted as due to the adsorption [29], in this case of 3,7-dimethylquinoxalin-2-(1H)-one, on the electrode surface. The double layer formed at the electrode-solution interface is considered as an electric capacitor, whose capacitance decreases due to the displacement of water molecules and other ions originally adsorbed on the electrode by the quinoxaline molecules, forming a protective film. The thickness of the film formed increases with increasing concentrations of the inhibitor, since more 3,7-dimethylquinoxalin-2-(1H)-one adsorbs on the surface, resulting in lower C_{dl} values. The results obtained are shown in Table 2. It can be clearly seen that the R_{ct} value increases with the inhibitor concentration, leading to an increase in the corrosion inhibition efficiency. The IE% values calculated from the EIS values are in complete agreement with those obtained from the Tafel, polarization resistance. The slight discrepancy in the IE% values obtained from different techniques can be interpreted as the result of different measurement times [32]. Therefore, these results suggest, once again, the formation of an insoluble inhibitor film due to the adsorption of 3,7-dimethylquinoxalin-2-(1H)-one onto the carbon steel surface.

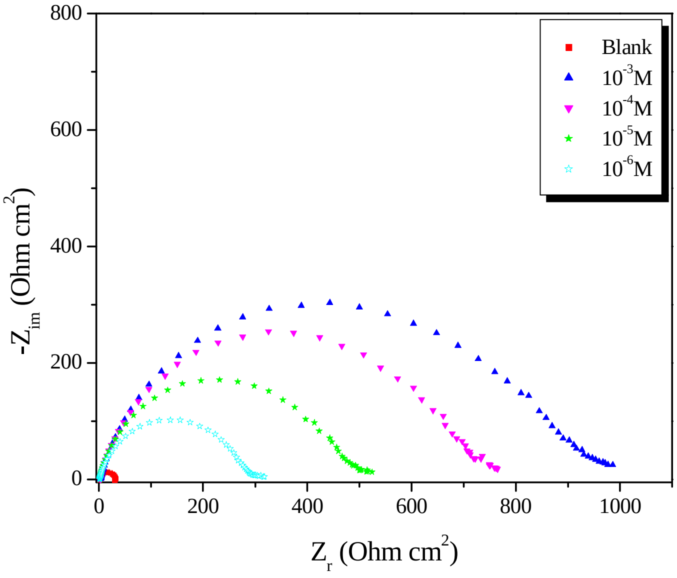

Figure 3. Nyquist diagrams for carbon steel in 1.0 M HCl containing different concentrations of DQO at 308 K.

Adsorption isotherms

Surfactants inhibit the corrosion of carbon steel by adsorption on the metal-solution interface. The adsorption provides the information about the interaction among the adsorbed molecules themselves as well as their interaction with the electrode surface [34,35]. The degrees of surface coverage (θ) for different concentrations of inhibitor in acidic media have been evaluated from electrochemical impedance spectroscopy by using the following equation: $\eta_z\%/100$ (Table 2).

Table 2 Electrochemical impedance parameters for carbon steel in 1.0 M HCl in absence and presence different concentrations of inhibitor

Inhibitor	Conc (M)	Rs (Ω cm^2)	R_{ct} (Ω cm^2)	f_{max} (Hz)	C_{dl} (μF/cm^2)	η_Z (%)	θ
Blank	1.0	1.67	031.0	63.34	80.99	-	-
DQO	10^{-3}	3.19	958.1	6.33	26.26	96.8	0.968
	10^{-4}	2.46	745.1	7.93	26.91	95.8	0.958
	10^{-5}	1.92	502.6	10.0	31.68	93.8	0.938
	10^{-6}	3.26	299.0	12.5	42.60	89.6	0.896

Attempts were made to fit these θ values to various isotherms including Frumkin, Langmuir, and Temkin. According to these isotherms, θ is related to the inhibitor concentration (C_{inh}):

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K_{\text{ads}}} + C_{\text{inh}} \qquad (5)$$

A straight line is obtained on plotting C_{inh}/θ vs. C_{inh} as shown in Fig. 4. The linear correlation coefficient (R^2) is almost equal to 1 (and the slope is very close to 1, indicating the adsorption of synthesized inhibitor on the carbon steel surface obeys the Langmuir adsorption isotherm. The strong correlation of the Langmuir adsorption isotherm may confirm the validity of this approach. The equilibrium constant (K_{ads}) for the adsorption-desorption process of tested inhibitor can be calculated from reciprocal of the intercept. The adsorptive equilibrium constant (K_{ads}) values are listed in Table 3. It is clear that, the large values indicate a strong adsorption of the synthesized inhibitor on the surface of carbon steel in 1.0 M HCl.

The free-energy of adsorption (ΔG°_{ads}) was calculated from the slope of the Langmuir isotherm, which showed the best correlation with the experimental data, according to the equation:

$$K_{ads} = (\frac{1}{55.5})\exp(-\frac{\Delta G^{\circ}_{ads}}{RT}) \qquad (6)$$

where R is gas constant and T is absolute temperature of experiment and the constant value of 55.5 is the concentration of water in solution in mol L^{-1} [37].

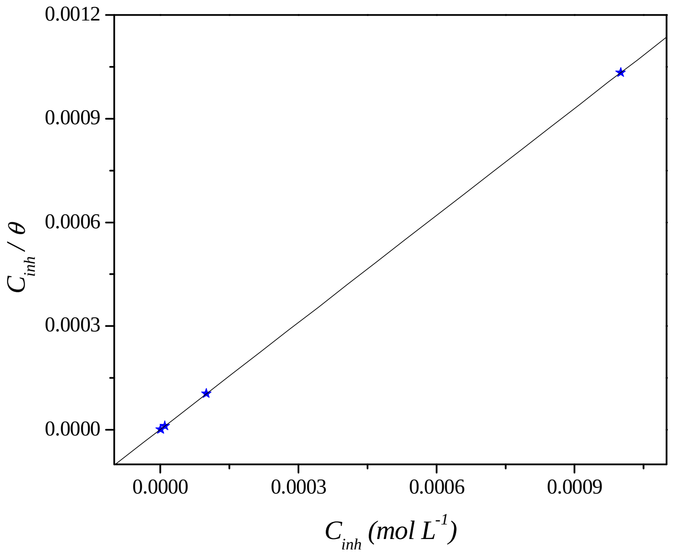

Figure 4. Langmuir adsorption of DQO on the carbon steel surface in 1.0 HCl solution.

Table 3 Thermodynamic parameters for the adsorption of DQO in 1.0 M HCl on the carbon steel at 308K.

Inhibitor	Slope	K_{ads} (M^{-1})	R^2	ΔG°_{ads} (kJ/mol)
DQO	1.03	2029830.39	1	-47.47

Generally, ΔG°_{ads} values of -20 kJ mol^{-1} or higher are associated with an electrostatic interaction between charged molecules and charged metal surface, physisorption; those of -40 kJ mol^{-1} or lower involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate covalent bond, chemisorption [38]. The value of ΔG°_{ads} is equal to -47.47 kJ mol^{-1}, the high value of ΔG°_{ads} shows that in the presence of 1.0 M HCl chemisorption of DQO may occur.

CONCLUSION

The results obtained show that 3,7-dimethylquinoxalin-2-(1H)-one is a good corrosion inhibitor for carbon steel under acidic conditions. The maximum inhibition efficiency was 98%. Excellent agreement between the inhibition efficiencies calculated using different techniques was obtained. The adsorption of the organic inhibitor onto the carbon steel surface was characterized by the decrease in (i) the cathodic and anodic current densities observed in the potentiodynamic polarization curves carried out in the presence of 3,7-dimethylquinoxalin-2-(1H)-one, (ii) the double-layer capacitance computed from electrochemical impedance spectroscopy experiments. The former effect was demonstrated by the decrease in the cathodic current densities observed in the potentiodynamic polarization curves without changes in the respective Tafel slope. The chemisorption aspect of the adsorption process was well-established by the value of ΔG°_{ads} = -47.47 kJ mol^{-1} calculated from the slope of the Langmuir isotherm, which best fitted the experimental data of the relationship between 3,7-dimethylquinoxalin-2-(1H)-one concentration and surface coverage.

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