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Anti-corrosion properties of indole derivative for carbon steel in HCl solution

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ABSTRACT

Weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) were used to investigate the corrosion inhibition of 1-acetylidoline-2,3-dione (Ind2) for carbon steel in hydrochloric acid. It is found that the inhibition efficiency of Ind2 increases with the increase of the concentration of Ind2. The results show that Ind2 possesses excellent inhibiting effect for the corrosion of carbon steel and the inhibitor acts as mixed-type inhibitor. The adsorption process is spontaneous and follows Langmuir adsorption isotherm model. The surface adsorption of the inhibitor molecules decreases the double-layer capacitance and increases the polarization resistance. The results obtained from the different techniques were in good agreement which prove the validity of these tools in the measurements of the tested inhibitor.

Keywords: Acid inhibitor, Carbon steel, EIS, Polarization, Langmuir.

INTRODUCTION

Corrosion is a fundamental process playing an important role in economics and safety, particularly for metals. The use of inhibitors is one of the most practical methods for protection against corrosion, especially in acidic media [1]. Most well-known acid inhibitors are organic compounds which have π bonds and contains hetero atoms such as sulphur, nitrogen and oxygen which allows the adsorption of compounds on the metal surface [2-25]. Furthermore, organic inhibitor adsorption on metal surface is influenced by organic inhibitor nature, surface charges on the metal, the type of aggressive solution, and the interaction of inhibitor with the metal surface. In hydrochloric acid solution, the metal surface due to its dissolution and organic inhibitor which is protonated carries positive charge [26]. The protonated organic inhibitor would be less adsorbed onto the metal surface leading to lower inhibition efficiencies due to the electrostatic interaction of positive charge.

The present paper describes a study of corrosion protection of 1-acetylidoline-2,3-dione (Ind2) on carbon steel in 1.0 M HCl using chemical (weight loss) and electrochemical techniques (EIS, potentiodynamic polarization). This inhibitor is of particular interest because of their high solubility in water, containing π -electrons and electronegative

atoms such as N & O in their molecules. These factors favour the interaction of this indole derivative with the metal. As far as we know no concrete report has been published so far for this compound in 1.0 M HCl with use of electrochemical and chemical techniques, hence the present study. The structure of these drugs is shown in the Figure 1.

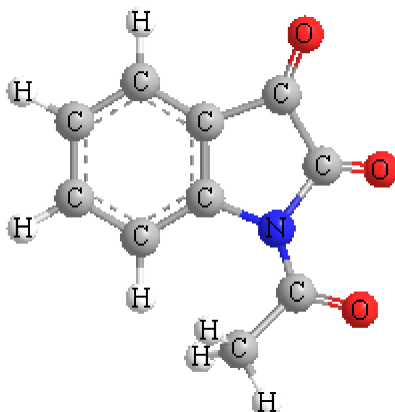


Figure 1. The chemical structure of the studied acridin derivative compound

MATERIALS AND METHODS

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 organic compound tested is 1-acetylidole-2,3-dione (Ind2). The concentration range of this compound was 10^{-3} to 10^{-6} M.

Weight loss measurements

Coupons were cut into $2 \times 2 \times 0.08$ cm³ dimensions are used for weight loss measurements. Prior to all measurements, the exposed area was mechanically abraded with 180, 320, 800, 1200 grades of emery papers. The specimens were washed thoroughly with bidistilled water, degreased and dried with ethanol. Gravimetric measurements are carried out in a double walled glass cell equipped with a thermostated cooling condenser. The solution volume is 50 mL. The immersion time for the weight loss is 6 h at 308 K. In order to get good reproducibility, parallel triplicate experiments were performed and the average weight loss value of three parallel carbon steel sheets was obtained. The corrosion rate (V) was calculated by the following equation:

$$V = \frac{w}{St} \quad (1)$$

Where V was the corrosion rate in (mg cm⁻² h⁻¹), w is the average weight loss of three parallel carbon steel sheets (mg), S was the total area of one carbon steel sheet (cm²), and t was immersion time (h).

With the calculated corrosion rate, the inhibition efficiency (η_{WL} %) was obtained as the following equation:

$$\eta_{WL} \% = \frac{V_0 - V}{V_0} \times 100 \quad (2)$$

Where V_0 and V are the values of corrosion rate without and with different concentration of inhibitor, respectively.

Polarization measurements

Electrochemical impedance spectroscopy

The electrochemical measurements were carried out using Volta lab (Tacussel- Radiometer PGZ 100) potentiostat and controlled by Tacussel corrosion analysis software model (Voltmaster 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 0.094 cm². 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 [27]:

$$\eta_z \% = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \times 100 \quad (3)$$

where R_{ct} and $R_{ct(inh)}$ were the values of polarization resistance in the absence and 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 -730 to -150 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:

$$I = I_{corr} \left[\exp\left(\frac{2.3\Delta E}{\beta_a}\right) - \exp\left(\frac{2.3\Delta E}{\beta_c}\right) \right] \quad (4)$$

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

$$\eta_{Tafel} \% = \frac{I_{corr}^{\circ} - I_{corr}^i}{I_{corr}^{\circ}} \times 100 \quad (5)$$

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

RESULTS AND DISCUSSION

Electrochemical Impedance Spectroscopy (EIS)

EIS technique was applied to investigate the electrode/electrolyte interface and corrosion processes that occur on carbon steel surface in presence and absence of Ind2. To ensure complete characterization of the interface and surface processes, EIS measurements were made at OCP in a wide frequency range at 308 K. Figure 2 shows Nyquist plots for carbon steel electrode immersed in 1.0 M HCl solution at 308 K in absence and presence of various concentrations of Ind2 at the respective open circuit potential. It is cleared from the figure 2 that the diameter of the semicircle increases with the increase in inhibitor concentration in the electrolyte, indicating an increase in corrosion resistance of the material [28].

The value of electrochemical double layer capacitance (C_{dl}) was calculated at the frequency, f_{max} using the following equation [29]:

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}} \quad (6)$$

where f_{max} is the frequency at which the imaginary component of the impedance is maximal.

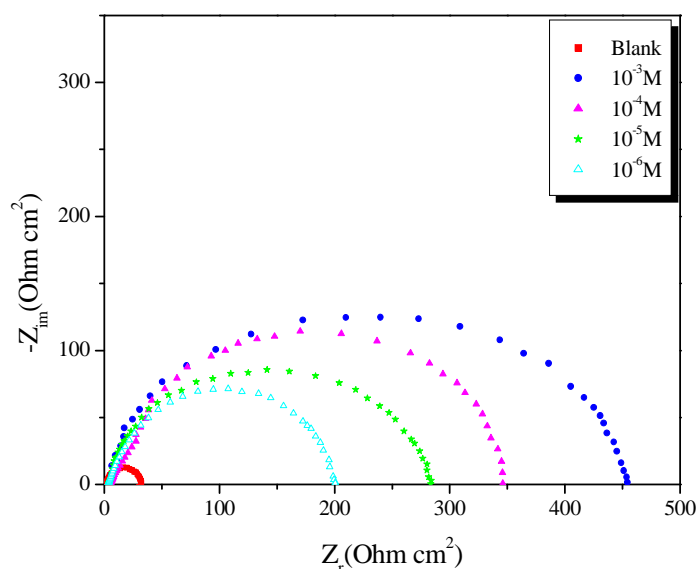


Figure 2. Nyquist plots for carbon steel in 1.0 M HCl solution in presence of various concentrations of Ind2 at 308K.

It is observed that addition of inhibitor increases the values of R_{ct} and reduces the C_{dl} value. The decrease in C_{dl} is due to increase in thickness of the electronic double layer [30]. The increase in R_{ct} values is due to the formation of protective film on the metal/solution interface [31,32]. This observation suggests that indole molecules function by adsorption on metal surface and thereby causing the decrease in C_{dl} values and increase in R_{ct} values. The charge transfer resistance (R_{ct}) values and the interfacial double layer capacitance (C_{dl}) values calculated from the curves are shown in the table 1.

Table 1. Electrochemical impedance parameters and inhibition efficiency for carbon steel in 1.0 M HCl solution with Ind2 at 308K.

Inhibitor	Conc (M)	R_{ct} (Ω cm ²)	f_{max} (Hz)	C_{dl} (μ F/cm ²)	η_z (%)
Blank	1	31.04	63.34	80.99	----
	10^{-3}	453.31	10.00	35.11	93.1
	10^{-4}	344.33	12.50	36.97	90.9
Ind2	10^{-5}	281.99	12.50	45.17	88.9
	10^{-6}	200.00	15.82	50.33	84.5

Potentiodynamic polarization curves

Potentiodynamic polarization curves for carbon steel in 1.0 M HCl solutions in the absence and presence of various concentrations of Ind2 at 308 K are shown in figure 3. The extrapolation of Tafel straight line allows the calculation of the corrosion current density (I_{corr}). The values of I_{corr} , the corrosion potential (E_{corr}), cathodic Tafel slopes (β_c) and the percentage of inhibition efficiency (η_{Tafel} %) are given in the table 2. It is evident from the figure that cathodic Tafel slopes (β_c) remain almost unchanged with increasing inhibitor concentration. This indicates that hydrogen evolution is activation controlled and the addition of inhibitor did not change the mechanism of cathodic hydrogen evolution reaction [33,34].

It is observed that the inhibition efficiency increased with increasing Ind2 concentration and exhibited both cathodic and anodic inhibition through adsorption on the carbon steel surface blocking active sites [35]. There is no definite change observed in the corrosion potential (E_{corr}). According to Riggs [36] and others if the displacement in E (i) is > 85 mV with respect to E , the inhibitor can be seen as a cathodic or anodic type, (ii) if displacement in E is < 85 , the inhibitor can be seen as mixed type. In our study the maximum displacement is less than 85, which indicates that Ind2 is a mixed type inhibitor. It is evident from the data that inhibition efficiency (η_{Tafel} %) increases with increase in concentration of the inhibitor. The corrosion current density (I_{corr}) decreases with increase in inhibitor concentration. The maximum inhibition efficiency of 79.8% is obtained at 10^{-6} M solution of Ind2.

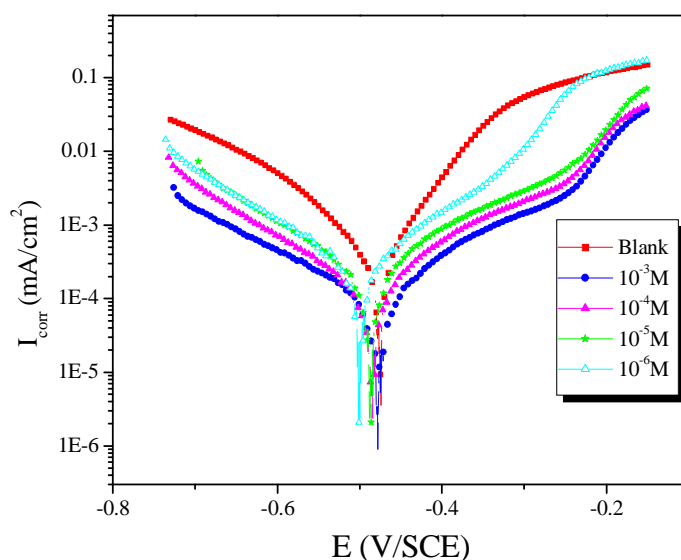


Figure 3. Polarisation curves of carbon steel in 1.0 M HCl for various concentrations of Ind2.

Table 2. Polarisation data of carbon steel in 1.0 M HCl without and with addition of inhibitor at 308 K.

Inhibitor	Conc (M)	$-E_{\text{corr}}$ (mV/SCE)	$-\beta_c$ (mV/dec)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	η_{Rafel} (%)
Blank	1.0	475.9	175.6	1077.8	----
Ind2	10^{-3}	477.3	167.3	88.9	91.7
	10^{-4}	486.2	154.1	116.9	89.1
	10^{-5}	485.4	161.8	163.0	84.9
	10^{-6}	500.0	157.5	217.9	79.8

Weight loss studies

The weight loss of carbon steel specimen in 1.0 M HCl solution, with and without different concentrations from the 1-acetylindoline-2,3-dione (Ind2), was determined after 6 h of immersion at 308 K. Obtained values of the inhibition efficiency (η_{WL} %) are given in Table 3. The inhibitor's presence reduces the corrosion rate of steel in hydrochloric acid. The inhibitory efficiency increases with the Ind2 concentration to reach 94.6% at 10^{-3} M, indicating that the indole compound tested is a good inhibitor of carbon steel in HCl solution. The corrosion inhibition is essentially due to the presence of electron donors groups (O, N) in the molecular structure of Ind2.

Table 3. The values of inhibition efficiency obtained from the weight loss for Ind2 at different concentration in 1.0 M HCl and at 308K.

Inhibitor	Conc (M)	v ($\text{mg cm}^{-2} \text{h}^{-1}$)	η_{WL} (%)	θ
Blank	1.0	1.070	-----	-----
Ind2	10^{-3}	0.058	94.6	0.946
	10^{-4}	0.084	92.1	0.921
	10^{-5}	0.112	89.5	0.895
	10^{-6}	0.170	84.1	0.841

Adsorption isotherm studies

The primary step in the action of inhibitors in acid solution is generally agreed to be the adsorption on the metal surface. This involves the assumption that the corrosion reactions are prevented from occurring over the area (or active sites) of the metal surface covered by adsorbed inhibitor species, whereas these corrosion reactions occur normally on the inhibitor-free area [37]. Accordingly, the fraction of surface covered with inhibitor species ($\theta = \eta_{\text{WL}} \% / 100$) can follow as a function of inhibitor concentration and solution temperature. The surface coverage (θ) data are very useful on discussing the adsorption characteristics. When the fraction of surface covered is determined as a function of the concentration at constant temperature, adsorption isotherm could be evaluated at equilibrium condition. The dependence of the fraction of the surface covered θ on the concentration C_{inh} of the inhibitor was tested graphically by fitting it to Langmuir's isotherm, which assumes that the solid surface contains a

fixed number of adsorption sites and each site holds one adsorbed species. Fig. 4 shows the linear plots for C_{inh}/θ versus C_{inh} , suggesting that the adsorption obeys the Langmuir's isotherm:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (7)$$

where C_{inh} is the inhibitor concentration, and K_{ads} the adsorptive equilibrium constant, representing the degree of adsorption (i.e., the higher value of K_{ads} indicates that the inhibitor is strongly adsorbed on the metal surface); the value of K_{ads} obtained from the reciprocal of intercept of Langmuir plot lines and the slope of these lines is near unity, meaning that each inhibitor molecule occupies one active site on the metal surface. The correlation coefficient (R^2) was used to choose the isotherm that best fit experimental data (Table 4).

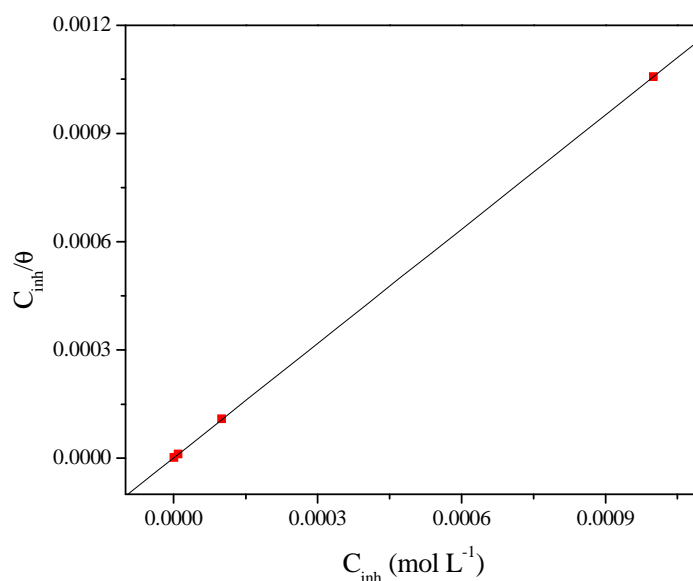


Figure 4. Langmuir adsorption of Ind2 on the carbon steel surface in 1.0 HCl solution.

From the intercepts of the straight lines on the C_{inh}/θ -axis (Figure 3), K_{ads} can be calculated which is related to free energy of adsorption, ΔG_{ads}° as given by

$$\Delta G_{ads}^{\circ} = -RTL \ln(55.5 K_{ads}) \quad (8)$$

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} .

Table 4. Thermodynamic parameters for the adsorption of Ind2 in 1.0 M HCl on the carbon steel at 308K.

Inhibitor	Slope	$K_{ads} (\text{M}^{-1})$	R^2	$\Delta G_{ads}^{\circ} (\text{kJ/mol})$
Ind2	1.05	846611.02	1	-45.24

Generally, the energy values of -20 kJ mol^{-1} or less negative are associated with an electrostatic interaction between charged molecules and charged metal surface, physisorption; those of -40 kJ mol^{-1} or more negative involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate covalent bond, chemisorption [38,39]. The value of ΔG_{ads}° is equal to $-45.24 \text{ kJ mol}^{-1}$. The large value of ΔG_{ads}° and its negative sign is usually characteristic of strong interaction and a highly efficient adsorption [40]. The high value of ΔG_{ads}° shows that in the presence of 1.0 M HCl chemisorption of Ind2 may occur. The possible mechanisms for chemisorption can be attributed to the donation of π -electron in the aromatic rings, the presence of one nitrogen and

three oxygen atoms in inhibitor molecule as reactive centers is an electrostatic adsorption of the protonated indole compound in acidic solution to adsorb on the metal surface.

CONCLUSION

The corrosion rate of carbon steel in HCl solution in the presence of Ind2 decreased with the increase in the surface coverage value, θ . Ind2 molecules adsorb on the carbon steel surface through chemisorptions interaction with the carbon steel surface. The negative values of free energy of adsorption ΔG_{ads}° , suggest the spontaneous adsorption of Ind2 on the carbon steel. Polarization studies reveal that Ind2 acts as a mixed type inhibitor. The surface adsorption of the used inhibitor led to a reduction in the double layer capacitance as well as an increase in the charge transfer resistance. The inhibitor efficiencies determined by chemical and electrochemical methods are in good agreement.

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