

Research Article

The Effect of New Pyridazinium-Based Ionic Liquid Derivative as Corrosion Inhibitor for Carbon Steel in 1M HCl Solution

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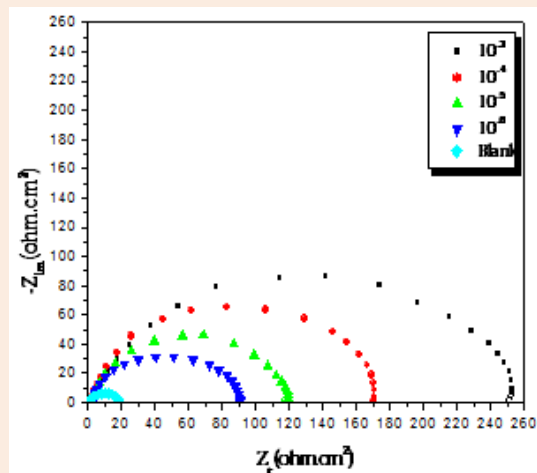
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Abstract

Eco-friendly ionic liquid 1-(2-ethoxy-2-oxoethyl)pyridazinium chloride (EOPC) newly synthesised was tested as corrosion inhibitor of carbon steel (CS) in normal hydrochloric acid solution using potentiodynamic polarisation electrochemical impedance spectroscopy (EIS) measurements and weight loss method. Gravimetric essays indicate that EOPC inhibits the corrosion of steel. The protection efficiency increases with increasing inhibitors concentration. That EOPC is a good inhibitor and inhibition efficiency reaches 92.80 % at the 10^{-3} M of EOPC. Potentiodynamic polarization curves showed that the EOPC affects both cathodic and anodic current and may be classified as mixed type inhibitors in 1M HCl. The adsorption of this compound on C-steel surface obeys Langmuir's adsorption isotherm.

Keywords: Carbon steel, Polarisation, EIS, ionic liquids, pyridazinium, Acid medium, Corrosion inhibition.



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Introduction

The inhibition of corrosion of carbon steel in acidic aqueous solutions by different organic compounds has been widely studied by several authors. The search for new and efficient corrosion inhibitors has become a necessity to protect metallic materials against corrosion [1]. The use of inhibitors is one of the most sensible methods for the protection of metals against corrosion in hydrochloric acid environment [2, 4]. Recently the research has been focused on the use of the Eco-friendly products or green inhibitors are known to have inhibitive action [3, 5–8]. In the last two decades, the organic compounds with low melting points which known as ionic liquid (IL), were considered as important topic of research in both industry and academia [4]. The unique properties of these materials help this family of organic compounds by changing the cations to find their smart applications. Many industrial applications of these ionic liquids have been found in organic synthesis, catalysis [9-15], chemical extraction [16, 17], electrochemistry [18-20], corrosion [21] and biochemistry [22]. But rare are the investigations on the voltammetric studies of IL reported in literature [23]. The inhibitory effect of Pyridazinium-based Ionic Liquid [24], azoles [25-33], and some ionic liquids derivatives such as imidazolium [34] is studied. Inhibitors, which reduce corrosion on metallic materials, can be divided into three kinds: (a) inorganic inhibitors, (b) organic inhibitors and (c) mixed material inhibitors [35]. The adsorption of inhibitors is influenced by the nature and surface charge of the metal, by the type of the aggressive electrolyte and by the chemical structure of the inhibitors. The principal types of

interaction between an organic inhibitor and a metal surface are physical adsorption and chemisorption. The adsorption of inhibitors is related to the presence of functional electronegative atoms as nitrogen, oxygen, sulphur and phosphorus or together with multiple bonds or aromatic rings in their molecular structure. This inhibitor are mainly used for acid inhibition processes [36–40]. Generally, the tendency to form a stronger coordination bond and, hence, to cause higher inhibition efficiency should follow the sequence $O < N < S < P$ [41]. In addition to this, structure and the lone pair of electrons found on the hetero atoms of the inhibitors play important characteristics that are used for determining the adsorption mechanism of these molecules on a metallic surface [42–46]. On the other hand, and despite the very interesting properties of ionic liquids (IL) [47–58] and their wide range of applications [59–63], these eco-friendly compounds have received little interest as corrosion inhibitors. It has been reported that some ILs based on imidazolium, pyridinium and pyridazinium exhibited corrosion inhibition properties for the corrosion of various metals [24, 64–73].

In the present work, the inhibition effects of a new pyridazinium– based ionic liquid (IL) compound, namely 1-(2-ethoxy-2-oxoethyl)pyridazinium chloride (*EOPC*) on the corrosion of C- steel in 1 M HCl would be studied, electrochemical behavior experiments were done in HCl media in the presence and absence of inhibitor using the weight loss method, potentiodynamic polarization and electrochemical impedance spectroscopic (EIS) studies. The effect of (IL) concentration on the corrosion inhibition efficiency will be examined. The chemical structures of the studied imidazolium ionic liquids are given in fig. 1.

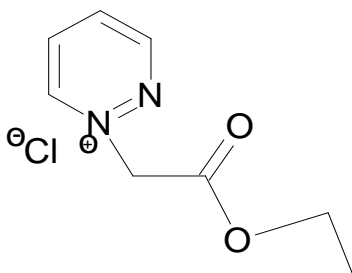


Figure 1 The chemical structure of the studied pyridazinium ionic liquid

Experimental

Materials

The steel used in this study is a carbon steel (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 solution (1M HCl) was prepared by dilution of analytical grade 37% HCl with double distilled water. The solution tests are freshly prepared before each experiment. The organic compound tested was ionic liquid 1-(2-ethoxy-2-oxoethyl) pyridazinium chloride (**EOPC**). The concentration range of this compound was 10^{-3} to 10^{-6} M. The studied organic compound was synthesized as described elsewhere [74].

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 80 mL. The immersion time for the weight loss is 6 h at 298 K.

Electrochemical impedance spectroscopy (EIS)

The electrochemical measurements were carried out using Volta lab (PGZ 100) potentiostat and controlled by 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. 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 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 298 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.

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 -800 mV to -200 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}).

Results and Discussion

Potentiodynamic polarization curves

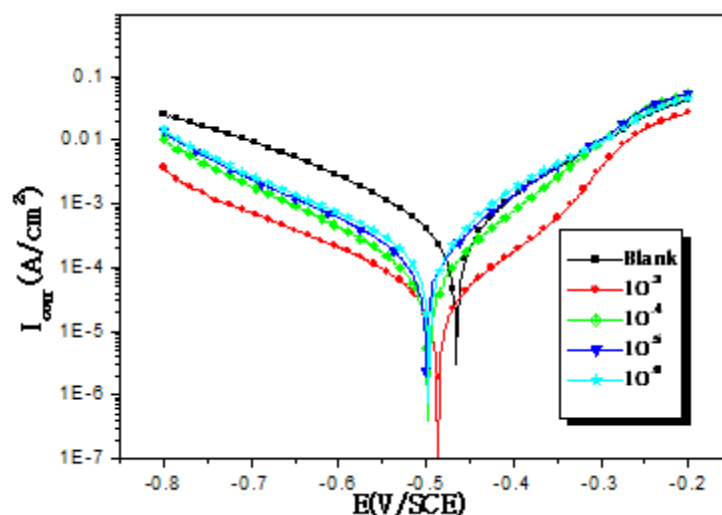


Figure 2 Potentiodynamic polarisation curves of C- Steel in 1M HCl in the presence of different concentrations of *EOPC*

The potentiodynamic polarization curves of C-Steel performed in 1M HCl in the absence and presence of different concentrations of *EOPC* at 298 K are presented in Fig.2. The potentiodynamic parameters such as corrosion potential (E_{corr}), cathodic Tafel slopes (b_c), corrosion current density (I_{corr}), were obtained from Tafel plots and the percentage inhibition efficiency values $IE(\%)$, were calculated using equation1:

$$IE \% = \frac{I_{corr} - I'_{corr}}{I_{corr}} \times 100 \quad (1)$$

Where, I_{corr} and I'_{corr} are uninhibited and inhibited corrosion current densities, respectively

The results are tabulated in table 1. From **Figure 2** and **table 1**, it can be seen that the addition of EOPC at all the studied concentrations resulted in the significant decrease in the corrosion current density (I_{corr}) and decrease in the corrosion rate, the presence of inhibitor molecule in the corrosive medium increases cathodic and anodic potentials. These changes increase with increase in inhibitor concentration. This behavior supports the adsorption of inhibitor on to C-steel surface and causes a barrier effect for mass and charge transfer for cathodic and anodic reactions.

Table 1 Corrosion parameters obtained from Tafel polarization studies in 1 M HCl medium at different concentrations of EOPC at 298 K

Inhibitor	$C_{inh}(M)$	$-E_{corr}(mV/SCE)$	$I_{corr}(\mu A/cm^2)$	$-b_c(mV/dec)$	IE(%)
Blank	0.00	480	588	168	-
EOPC	10^{-3}	486	48	182	91.84
	10^{-4}	498	92	161	84.35
	10^{-5}	501	136	157	76.87
	10^{-6}	494	180	155	69.39

As can be seen from table 1, E_{corr} values did not change significantly (only the displacement was < 35 mV) in presence of inhibitor, based on the marked decrease of the cathodic and anodic current densities upon introducing the inhibitor in the aggressive solution, EOPC can be considered as a mixed-type inhibitor, meaning that the addition of EOPC reduces the anodic dissolution and also retards the cathodic hydrogen evolution reaction, EOPC block the reaction sites of Carbon steel electrode. It is also evident that percentage efficiency of the inhibitor increased with increase in the concentration of the inhibitor. It can be observed that 'bc' values are slightly increased in the presence of inhibitor indicating that it can be mainly due a mixed type cathodic inhibitor [75, 76]. The inspection of results in table 1 indicate that EOPC inhibits the corrosion process in the studied range of concentrations and IE (%) increases with C_{inh} , reaching its maximum value, 91.84%, at 10^{-3} M. These polarization curves tests were in good agreement with the corrosion weight loss and impedance measurements.

Weight loss measurements and adsorption isotherm

Weight loss measurements were done according to the method described previously [77]. And is a non-electrochemical technique for the determination of corrosion rates and inhibitor efficiency which provides more reliable results than electrochemical techniques because the experimental conditions are approached in a more realistic manner yet the immersions tests are time-consuming [78-80]. Therefore, due to such differences (experimental conditions), the values would obviously differ from the electrochemical values. All the tests were conducted in aerated 1 M HCl at 298 K with different concentrations of EOPC and the Values of the inhibition efficiency and corrosion rate obtained from the weight loss measurements of C-steel for different concentrations of EOPC in 1M HCl at 298 K after 6h of immersion are given in table 2. At the end of the tests the specimen were carefully washed in acetone and then weighed. Duplicate experiments were performed in each and the mean value of the weight loss has been reported. The inhibition efficiency ($E_w\%$) and surface coverage (θ) were determined by using the following equations:

$$E_w \% = \frac{W_{corr} - W'_{corr}}{W_{corr}} \times 100 \quad (2)$$

$$\theta = 1 - \frac{W'_{corr}}{W_{corr}} \quad (3)$$

Where W_{corr} and W'_{corr} are the corrosion rates of C-steel due to the dissolution in 1M HCl in the absence and the presence of definite concentration of inhibitor respectively, and θ is the degree of surface coverage of the inhibitor.

Table 2 Weight loss data of mild steel in 1 M HCl for various concentration of the *EOPC*

Conc (M)	W'_{corr} (mg. cm ⁻² . h ⁻¹)	E_w (%)	θ
0.00	1.021	-	-
10 ⁻³	0.092	90.98	0.98
10 ⁻⁴	0.104	89.81	0.89
10 ⁻⁵	0.151	85.21	0.85
10 ⁻⁶	0.182	82.17	0.82

It can be seen from table 2, that inhibition *EOPC* inhibits the corrosion of C-steel and efficiency increases with the increasing inhibitors concentration. The maximum inhibition was observed around 92% at 298 K with respect to the *EOPC* concentration 10⁻³ M at 298 K and upon comparing the results of Weight loss measurements studies with the analogous pyridazinium based ionic liquid inhibitor from literature[74], the observed results are in good agreement with the *EOPC* inhibitor. In order to understand the mechanism of corrosion inhibition, the adsorption behaviour of the adsorbate on the carbon steel surface must be known. Two main types of interaction can describe the adsorption of the compounds of *EOPC*: physical adsorption and chemisorption. These are influenced by the chemical structure of the inhibitor, the type of the electrolyte and the charge and nature of the metal. The information on the interaction between the inhibitor molecules of *EOPC* and the metal surface can be provided by adsorption isotherm. The degree of surface coverage (θ) for different concentrations of inhibitor was evaluated from Weight loss measurements. Attempts were made to fit θ values to various isotherms including Frumkin, Temkin and Langmuir. It was found that the data best fit was obtained with the Langmuir isotherm **figure 3**. According to this isotherm θ is related to concentration inhibitor C via[81]:

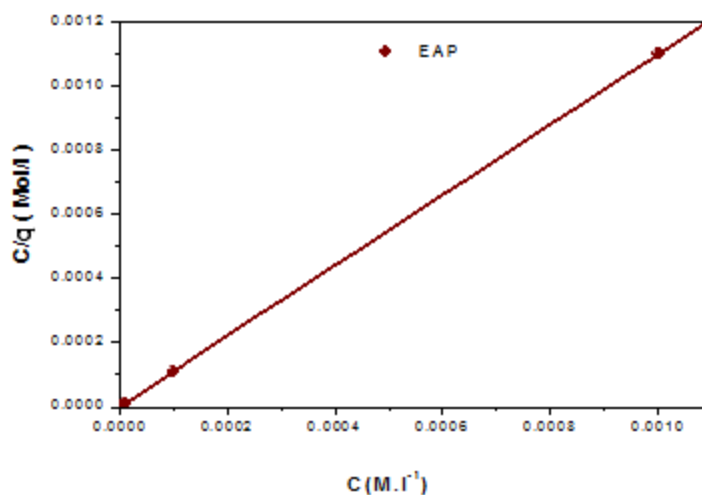


Figure 3 Plots of Langmuir adsorption isotherm of *EOPC* on the steel surface at 298K

$$\frac{C_{inh}}{\theta} = \frac{1}{K} + C_{inh} \quad (4)$$

Where K is the adsorption/desorption equilibrium constant, C_{inh} is the corrosion inhibitor concentration in the solution

$$\log K = -1,74 - \left(-\frac{\Delta G_{Ads}}{2,303RT} \right) \quad (5)$$

Where, ΔG_{Ads} is the free energy of adsorption,

It was found that figure 3 (plot of $\frac{\theta}{C}$ versus C) gives straight line with slope near to 1, indicating that the adsorption of compound under consideration on C-steel / acidic solution interface obeys Langmiur's adsorption. The free energy of adsorption (ΔG_{ads}) can be calculated from the K_{ads} value obtained from the above correlation:

$$\Delta G_{ads} = -RT \ln (55.5 \times K_{ads}) \quad (6)$$

Where, 55.5 is the concentration of water, R is the universal gas constant and T is the absolute temperature. The adsorption-desorption equilibrium constant K_{ads} , was determined as 10^{-3} M, leading to $\Delta G_{ads} = -44.81$ kJ.mol⁻¹ for *EOPC* at the temperature of 298 K. The negative sign of ΔG_{ads} indicates the spontaneity of the adsorption process and stability of the adsorbed layer on the electrode surface [82, 83]. Generally, the values of ΔG_{ads} around -20 kJ mol⁻¹ or less negative are known to be associated with physical adsorption (electrostatic interactions between the inhibitor and charged surface) while those around -40 kJ .mol⁻¹ or more negative is are known to be associated with chemisorption (charge sharing or transferring from organic molecules to the metal surface and form a coordinate type of metal bond) [84]. From this estimation, it can be concluded that the *EOPC* is chemically adsorbed on the charged C-steel surface thus creating a chemical interaction. A plausible explanation of this chemisorption in terms of corrosion mechanism would be the adsorption of *EOPC* molecules onto both anodic and cathodic sites of the corroding carbon steel surface [85]. The adsorption on the cathodic site would result from the existence of the *EOPC* as cationic species which is in fact very obvious for the investigated ionic liquid. This will hinder to some extent the hydrogen evolution reaction. While, the adsorption of this compound onto the anodic sites would be driven by the lone electron pairs of nitrogen and the π -electrons of C=N and C=C, which will causes a downturn of dissolution of carbon steel [73].

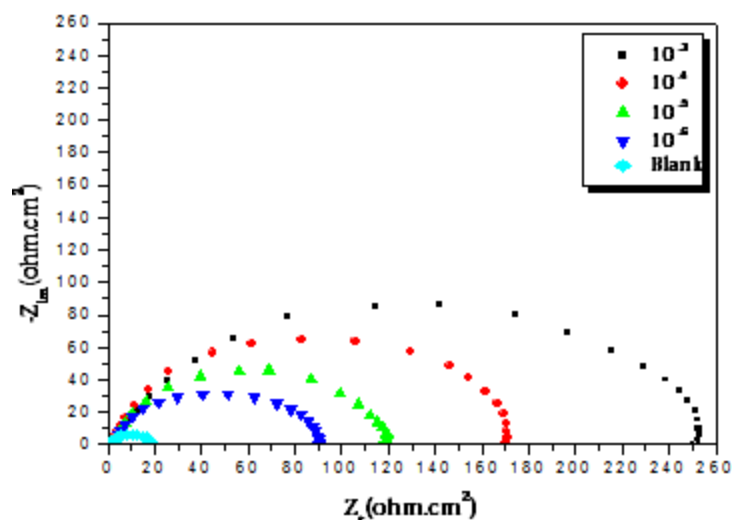


Figure 4 Nyquist plots for carbon steel in 1 M HCl containing different concentrations of *EOPC*

Electrochemical impedance spectroscopy (EIS)

The representative Nyquist plot of C-stel in 1.0 M HCl solutions in the absence and presence of different concentrations of *EOPC* at room temperature after 30 min of immersion are given in fig. 4. It shows the variation of charge transfer resistance (R_{ct}) with the increase in concentration.

As observed, the Nyquist plots contain a depressed semi-circle with the center below the real X-axis, which is size increased by increasing the inhibitor concentrations, indicating that the corrosion is mainly a charge transfer process [82, 86] and the formed inhibitive film was strengthened by the addition of *EOPC*. The depressed semi-circle is the characteristic of solid electrodes and often refers to the frequency dispersion which arises due to the roughness and other inhomogeneities of the surface [87].

The charge transfer resistance values R_{ct} and double-layer capacitance values C_{dl} are shown in **table 3**. The inhibition efficiency $E_{Rt}\%$ was calculated by the charge transfer resistance as follows:

$$E_{Rt} \% = \frac{R'_{ct} - R_{ct}}{R'_{ct}} \times 100 \quad (7)$$

Where, R_{ct} and R'_{ct} are the charge transfer resistance values without and with inhibitor respectively. Values of the charge transfer resistance R_{ct} were obtained from these plots by determining the difference in the values of impedance at low and high frequencies as suggested by [88].

Table 3 Electrochemical Impedance parameters for corrosion of steel in acid medium at various contents of *EOPC*

	$C_{inh}(M)$	$R_{ct}(\Omega.cm^2)$	$f_{max}(Hz)$	$C_{dl}(\mu F.cm^{-2})$	$E_{Rt}(\%)$
Blank	0.00	18	40	221.16	-
	10^{-3}	250	10	63.69	92.80
EOPC	10^{-4}	168	10	94.78	89.28
	10^{-5}	118	16	84.34	84.74
	10^{-6}	91	16	109.36	80.22

The inhibition efficiencies, calculated from impedance results, show the similar trend to those obtained from potentiodynamic polarization measurements and the maximum percentage was achieved at the concentration of 10^{-3} M (92.80%). It can be seen that the presence of *EOPC* concentrations increases the values of R_{ct} and reduce the C_{dl} values. The decrease in C_{dl} , which can result from a decrease in local dielectric constant and / or an increase in the thickness of the electric double layer [89], Furthermore, the decreased values of C_{dl} may be due to the replacement of water molecules at the electrode interface by the organic inhibitor of lower dielectric constant through adsorption [90], suggested that *EOPC* molecules function by adsorption at the metal / solution interface. The Nyquist diagrams were analyzed in terms of the equivalent circuit of the electrical double layer as shown in fig. 5, where R_e represents the electrolyte resistance and the electrical leads [91], R_{ct} the charge transfer resistance. Due to the dispersing effect resulted from the inhomogeneities and other roughness of the electrode surface [92-94], the impedance loops measured are depressed semi-circles with their centers below the real axis, so one constant phase element (CPE) is determined for the capacitive element to give a more specific fit in **figure 4**.

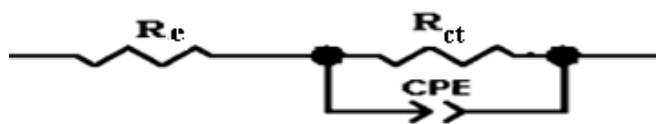


Figure 5 Equivalent circuit model used to fit the impedance spectra for the studied system.

The size of the semi circle increased with increasing *EOPC* concentration without changing the trend indicates the similar charge transfer process of the corrosion. Since, depression in Nyquist plot was observed, the constant phase element (CPE) is introduced in the circuit instead of pure double layer capacitor[95]. A similar circuit has been described in the literature for the acidic corrosion inhibition of steel [96].

Conclusions

In the present study, the inhibitive effect of Eco-friendly ionic liquid 1-(2-ethoxy-2-oxoethyl) pyridinium chloride (*EOPC*) inhibitor in this method show the good efficiency against the corrosion inhibition over C-steel. The *EOPC* act as a mixed inhibitor and its inhibition efficiency increases with increasing inhibitor concentration and reaches 92.80% at $10^{-3}M$. Weight loss, Tafel polarization and electrochemical impedance studies were performed to demonstrate the inhibition behavior of *EOPC* and the inhibition efficiencies obtained from all these methods were found to be in good agreement with each other. The negative values of ΔG_{ads} indicate spontaneous adsorption of the inhibitor on the surface of steel. The inhibitor acts by being adsorbed on C-steel surface according to classical Langmuir adsorption model.

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