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Corrosion and corrosion inhibition of carbon steel in hydrochloric acid solutions by 2-[Bis-(3,5-dimethyl-pyrazol-1-ylmethyl)-amino]-3-hydroxy-butyric acid

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ABSTRACT

A new corrosion inhibitor namely 2-[Bis-(3,5-dimethyl-pyrazol-1-ylmethyl)-amino]-3-hydroxy-butyric acid (Bip2) has been synthesized and its inhibitive performance towards the corrosion of carbon steel in 1.0 M hydrochloric acid has been investigated. Corrosion inhibition was studied by chemical method (weight loss) and electrochemical techniques include Tafel extrapolation method and electrochemical impedance spectroscopy (EIS). The efficiency of the inhibitor increases with increase in the inhibitor concentration. Results obtained reveal that the used Bipyrazole derivative performs as corrosion inhibitor for carbon steel in 1.0 M HCl. Double layer capacitance, C_{dl}*, and charge transform resistance,* R_{ct} *values were derived from Nyquist plots obtained from A.C. impedance studies. Changes in impedance parameters are indicative of the adsorption of this inhibitor on the steel surface. Polarisation studies showed that Bip2 was mixed-type inhibitor in 1.0 M HCl. The values of the inhibition efficiency calculated from the three techniques are in reasonably good agreement. The adsorption of this compound on carbon steel surface is found to obey Langmuir adsorption isotherm.*

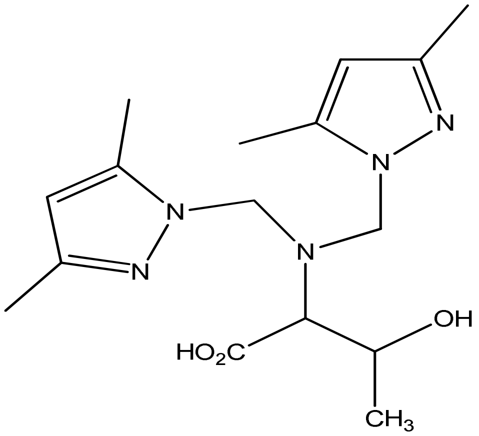
Keywords: Bipyrazole derivative, Carbon steel, Tafel, EIS, Weight loss.

INTRODUCTION

Steel is the most important engineering and construction material in the world. It is used in every aspect of our lives, from automotive manufacture to construction products, from steel toecaps for protective footwear to refrigerators and washing machines and from cargo ships to the finest scalpel for hospital surgery. There are several thousands steel grades published, registered, or standardized worldwide, all of which have different chemical compositions, and special numbering systems have been developed in several countries to classify the huge number of alloys. In addition, all the different possible heat treatments, microstructures, cold-forming conditions, shapes, and surface finishes mean that there are an enormous number of options available to the steel user. Fortunately, steels can be classified reasonably well into a few major groups according to their chemical compositions, applications, shapes, and surface conditions.

Corrosion problems have received a considerable amount of attention because of their attack on materials. The use of inhibitors is one of the most practical methods for protection against corrosion. Several works have studied the influence of organic compounds containing nitrogen on the corrosion of steel in acidic media [1-21], most organic inhibitors act by adsorption on the metal surface [22]. This phenomenon is influenced by the nature and surface charge of metal, type of aggressive medium and chemical structure of inhibitors. The adsorption of corrosion inhibitor depends mainly on physico-chemical properties of the molecule such as functional groups, steric factor, molecular size, molecular weight, molecular structure, aromaticity, electron density of the donor atoms and π-orbital character of donating electrons [23-27] and also on the electronic structure of the molecules [28,29].

The aim of the present study is to determine the inhibition efficiency of 2-[Bis-(3,5-dimethyl-pyrazol-1-ylmethyl)-amino]-3-hydroxy-butyric acid (Bip2) for the corrosion of carbon steel in HCl medium containing different concentration of inhibitor. The Molecular structure of Bip2 is shown as follows (scheme 1):



Scheme 1. Molecular structure of Bip2

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 2-[Bis-(3,5-dimethyl-pyrazol-1-ylmethyl)-amino]-3-hydroxy-butyric acid (Bip2). The concentration range of this compound was 10^{-3} to 10^{-6} M.

Weight loss measurements

Coupons were cut into 2 × 2 × 0.08 cm^3 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:

$$v = \frac{w}{St} \tag{1}$$

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 (cm^2), and t was immersion time (h).

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

$$\eta_{WL}\% = \frac{v_0 - v}{v_0} \times 100 \tag{2}$$

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) 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 0.094 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 (Eocp). After measuring the Eocp, 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 [30]:

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

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 -700 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}). From the polarization curves obtained, the corrosion current (I_{corr}) was calculated by curve fitting using the equation:

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

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

$$\eta_p \% = \frac{I^{\circ}_{corr} - I^{i}_{corr}}{I^{\circ}_{corr}} \times 100 \qquad (5)$$

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

RESULTS AND DISCUSSION

Weight loss measurements

Effect of inhibitor concentration

The weight-losses for steel in aerated molar HCl containing different concentrations of Bip2 at 308 K are given in Table 1. The inhibitor' presence reduces the corrosion rate of steel in hydrochloric acid. The inhibitory efficiency increases with the 2-[Bis-(3,5-dimethyl-pyrazol-1-ylmethyl)-amino]-3-hydroxy-butyric acid (Bip2) concentration to reach 95.9% at 10^{-3} M indicating that the Bipyrazole compound tested is a good inhibitor of steel in HCl solution. The corrosion inhibition is essentially due to the presence of electron donors groups (O, N) in the molecular structure of the Bipyrazole tested.

Figure 1 shows the variation of corrosion rate ν and of the inhibition efficiency (η_{WL} %) with the concentration of the additive. The curves obtained indicate that the η_{WL} % increases with increasing the concentration of the additive, but the corrosion rate decreases.

Table 1. The values of inhibition efficiency obtained from the weight loss for Bip2 at different concentration in 1.0 M HCl and at 308K.

Inhibitor	Conc (M)	ν (mg cm^{-2} h^{-1})	η_{WL} (%)	θ
Blank	1.0	1.070	-----	------
Bip2	10^{-3}	0.044	95.9	0.959
	10^{-4}	0.089	91.7	0.917
	10^{-5}	0.137	87.2	0.872
	10^{-6}	0.209	80.5	0.805

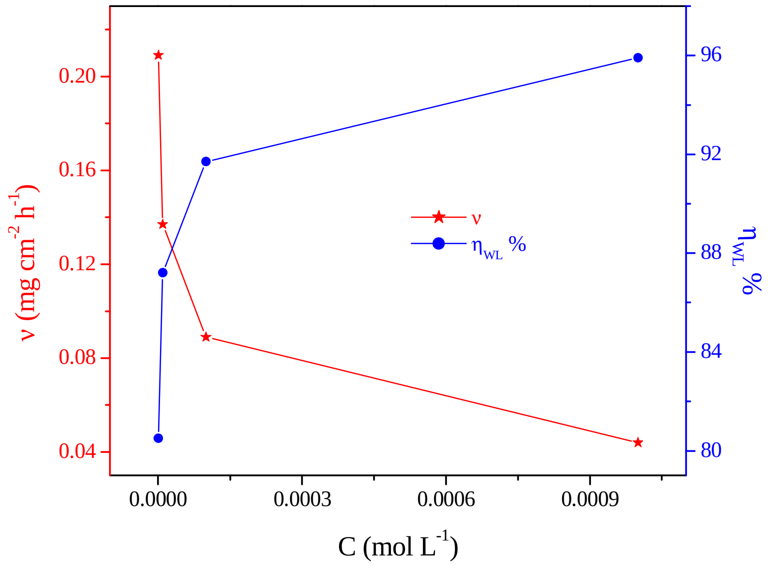


Figure 1. Variation of the corrosion rate and inhibitive efficiency against the Bip2 concentrations.

Adsorption isotherm

The adsorption on the corroding surfaces never reaches the real equilibrium and tends to reach an adsorption steady state. However, when the corrosion rate is sufficiently small, the adsorption steady state has a tendency to become a quasi-equilibrium state. In this case, it is reasonable to consider the quasi-equilibrium adsorption in a thermodynamic way using the appropriate equilibrium isotherms [31]. The efficiency of the Bipyrazole derivative as a successful corrosion inhibitor mainly depends on its adsorption ability on the metal surface. So, it is essential to know the mode of adsorption and the adsorption isotherm that can give valuable information on the interaction of inhibitor and metal surface. The surface coverage values, θ (θ = (η_{WL} %)/100), for different concentrations of Bipyrazole derivative were used to explain the best adsorption isotherm. A plot of C_{inh}/θ versus C_{inh} (Figure 2) gives a straight line with an average correlation coefficient of 0.9999 and a slope of nearly unity suggests that the adsorption of 2-[Bis-(3,5-dimethyl-pyrazol-1-ylmethyl)-amino]-3-hydroxy-butyric acid (Bip2) molecules obeys Langmuir adsorption isotherm, which can be expressed by the following equation:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \qquad (6)$$

where C_{inh} is the inhibitor concentration and K_{ads} is the equilibrium constant for adsorption-desorption process.

From the intercepts of the straight lines on the C_{inh}/θ-axis (Figure 2), K_{ads} can be calculated which is related to free energy of adsorption, ΔG°_{ads} as given by

$$\Delta G^{\circ}_{ads} = -RTLn(55.5K_{ads}) \qquad (7)$$

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}. The results are shown in Table 2.

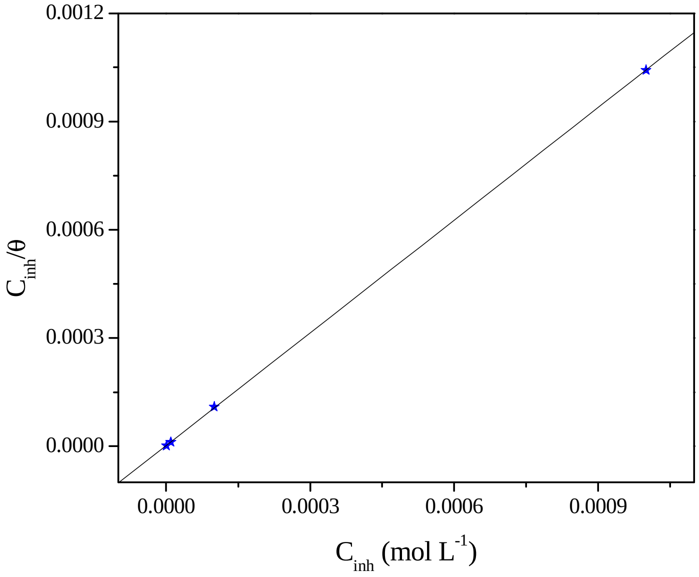


Figure 2. Langmuir adsorption of IBip2 on the carbon steel surface in 1.0 HCl solution.

Table 2. Some parameters from Langmuir model for carbon steel in 1.0 M HCl at 308K.

Inhibitor	Slope	K_{ads} (M^{-1})	R^2	ΔG°_{ads} (kJ/mol)
Bip2	1.04	507045.40	0.99999	-43.92

The negative values of ΔG°_{ads} calculated from Eq. (7), are consistent with the spontaneity of the adsorption process and the stability of the adsorbed layer on the carbon steel surface. Generally, values of ΔG°_{ads} up to -20 kJ mol^{-1} are consistent with physisorption, while those around -40 kJ mol^{-1} or higher are associated with chemisorption as a result of the sharing or transfer of electrons from organic molecules to the metal surface to form a coordinate bond [32]. In the present study, the calculated standard free energy of adsorption value is closer to -40 kJ mol^{-1} (Table 2). Therefore it can be concluded that the adsorption of the Bip2 on the carbon steel surface is more chemical than physical [33].

Potentiodynamic Polarization Measurements

Figure 3 showed potentiodynamic polarization curves for the carbon steel electrode in 1.0 M HCl solution with and without different concentrations of Bip2. It is clear that the current density decreases with the presence of Bipyrazole derivative; this indicated that Bip2 adsorbed on the metal surface, and hence inhibition occurs. Values of corrosion potential (E_{corr}) and corrosion current density (I_{corr}), obtained by extrapolation of the Tafel lines, cathodic Tafel slope (bc), and η_p (%) for different concentrations of Bip2 in 1.0 M HCl, are given in Table 3. The potentiodynamic curves show that there is a clear reduction of both the anodic and cathodic currents in the presence of Bip2 compared with those for the blank solution. It is clear that the cathodic reaction (hydrogen evolution) and the anodic reaction (dissolution metal) were inhibited. The values of cathodic Tafel slope bc in the presence of inhibitor was changing, which clearly indicates that the prepared compound influence the kinetics of hydrogen evolution reaction. This indicates an increase in the energy barrier for proton discharge leading to less gas evolution. The value of η_p (%) increased with increase in concentration of inhibitor, which indicates higher surface coverage of the metal.

It was found from the values in Table 3, the corrosion current density decreases with increase in treatment time and consistent increase of protection efficiency. The protection efficiency was increased with increase in concentration of inhibitor. The corrosion protection efficiency at higher concentration may be attributed to the formation of more stable film on the electrode surface. There is no definite trend observed in the E_{corr} values in the presence of Bip2. In literature [34], it has been reported that (i) if the displacement in E_{corr} is > 85 mV with respect to E_{corr}, the inhibitor can be seen as a cathodic or anodic type and (ii) if displacement in E_{corr} is < 85, the inhibitor can be seen as mixed type. In the present study, shift in E_{corr} values is in the range of 8-25 mV, suggesting that Bip2 acted as mixed type of inhibitor [34,35].

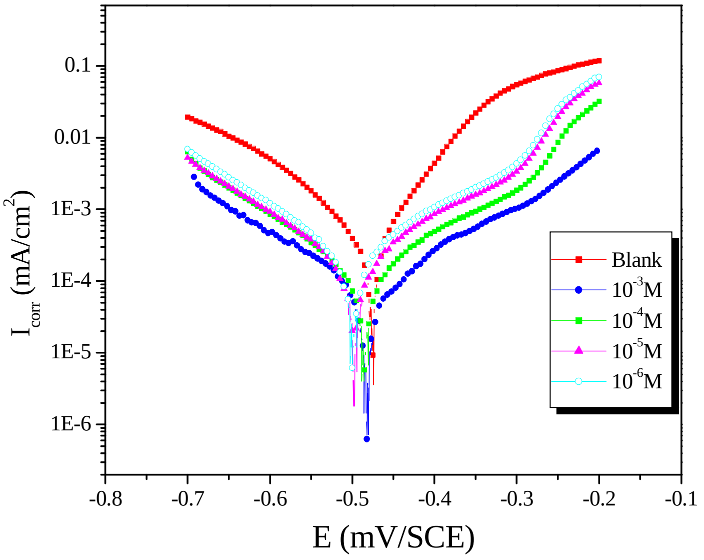


Figure 3. Polarisation curves of carbon steel in 1.0 M HCl for various concentrations of Bip2.

Table 3. Polarisation data of carbon steel in 1.0 M HCl without and with addition of inhibitor at 308 K.

Inhibitor	Conc (M)	$-E_{corr}$ (mV/SCE)	$-b_c$ (mV/dec)	I_{corr} (μA/cm^2)	η_p (%)
Blank	1.0	475.9	175.6	1077.8	----
Bip2	10^{-3}	483.6	140.1	66.3	93.8
	10^{-4}	486.6	132.7	112.0	89.6
	10^{-5}	495.8	139.3	157.3	85.4
	10^{-6}	501.4	138.6	228.6	78.8

Electrochemical Impedance Spectroscopy

Electrochemical impedance measurements were carried over the frequency range from 100 kHz to 0.01 Hz at open circuit potential. The simple equivalent Randle circuit for these studies is shown in Fig. 5, where R_e represents the solution resistance; the parallel combination of resister, R_t and capacitor C_{dl} represents the protective film/ metal interface. The impedance behavior of carbon steel in 1.0 M HCl with and without addition of various concentrations of Bip2 is presented as complex impedance plot (Nyquist plot) in Fig. 4. The existence of single semi circle shows the single charge transfer process during dissolution which is unaffected by the presence of inhibitor molecules. Deviation from perfect circular shape is often referred to the frequency dispersion of interfacial impedance. This anomalous behavior is generally attributed to the inhomogeneity of the metal surface arising from surface roughness or interfacial phenomena [37-39]. It is seen that addition of inhibitor increases the values of charge transfer resistance (R_{ct}) and reduces the double layer capacitance (C_{dl}). The decrease in C_{dl} is attributed to increase in thickness of electronic double layer [40]. The increase in R_{ct} value is ascribed to the formation of protective film on the metal/solution interface [41]. These observations suggest that Bip2 molecules function by adsorption at metal surface thereby causing the decrease in C_{dl} values and increase in R_{ct} values. The charge transfer resistance (R_{ct}), maximum frequency (f_{max}), the interfacial double layer capacitance (C_{dl}) and η_z (%) derived from these curves are given in Table 4.

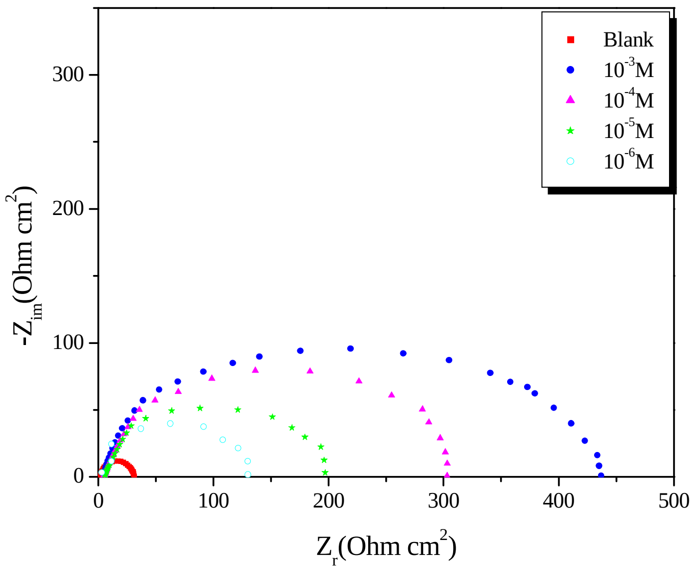


Figure 4. Nyquist plots for carbon steel in 1.0 M HCl solution in presence of various concentrations of Bip2 at 308K.

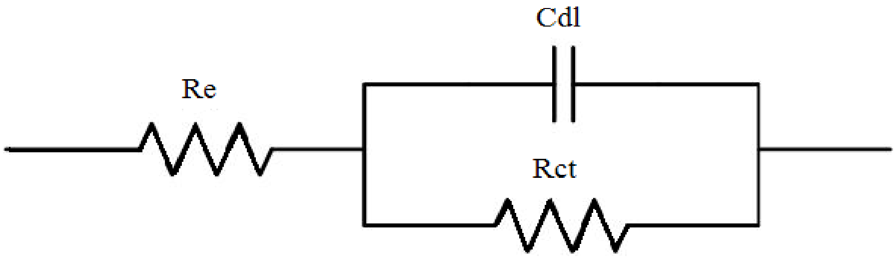


Figure 5. Electrical equivalent circuit (R_e: solution resistance; R_{ct}: charge transfer resistance; C_{dl}: double layer capacitance).

Table 4. Electrochemical impedance parameters and inhibition efficiency for carbon steel in 1.0 M HCl solution with Bip2 at 308K.

Inhibitor	Conc (M)	Re (Ω cm^2)	R_{ct} (Ω cm^2)	f_{max} (Hz)	C_{dl} (μF/cm^2)	η_z (%)
Blank	1.0	1.67	31.04	63.34	80.99	----
Bip2	10^{-3}	3.63	437.50	10.00	36.38	92.9
	10^{-4}	3.70	303.24	12.50	41.99	89.7
	10^{-5}	3.88	197.09	15.82	51.05	84.2
	10^{-6}	3.90	130.1	20.00	61.17	76.2

The double layer capacitance C_{dl} values for different inhibitor concentrations have been derived using the relation.

$$C_{dl} = \frac{1}{2\pi f_{\max} R_{ct}} \tag{8}$$

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

The thickness of the protective layer δ_{org} is related to C_{dl} by the following equation [42]:

$$C_{dl} = \frac{\varepsilon_r \varepsilon_0}{\delta_{org}} \tag{9}$$

where, ε_0 is the dielectric constant and ε_r is the relative dielectric constant. This decrease in the C_{dl} may result from a reduction in local dielectric constant and/or an increment in the thickness of the electrical double layer. The phenomenon proposed that Bip2 molecules function by adsorption at the metal/solution interface. Thus, the change in C_{dl} values was due to the gradual replacement of water molecules by the adsorption of the organic molecules on the metal surface, decreasing the magnitude of metal dissolution [43].

A comparison may be made between inhibition efficiency E (%) values obtained by different methods (weight loss, polarisation curves and EIS methods). Figure 6 shows a curve that compares the E (%) values obtained. One can see that whatever the method used, no significant changes are observed in E (%) values. We can then conclude that there is a good correlation with the three methods used in this investigation at all tested concentrations and that Bip2 is an efficient corrosion inhibitor.

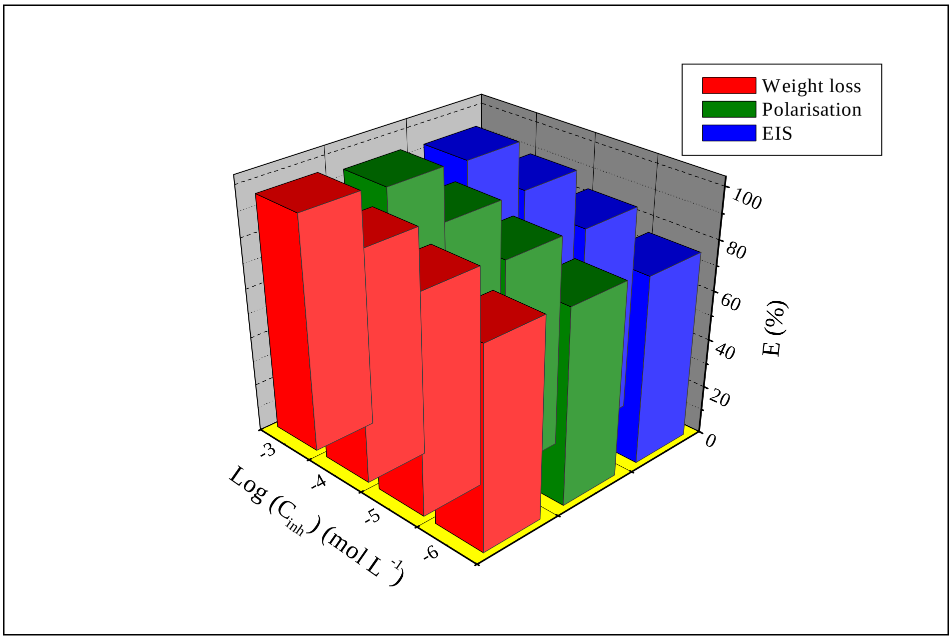


Figure 6. Comparison of inhibition efficiency (E %) values obtained by weight loss, polarisation and EIS methods.

CONCLUSION

2-[Bis-(3,5-dimethyl-pyrazol-1-ylmethyl)-amino]-3-hydroxy-butyric acid (Bip2) acts as a good inhibitor for the corrosion of carbon steel in 1.0 M HCl. The inhibition efficiency of Bip2 increases with increasing concentration of inhibitor. The adsorption of Bip2 molecules on carbon steel surface has been described by Langmuir adsorption isotherm. The high value of the adsorption equilibrium constant suggested that Bip2 was strongly adsorbed on the carbon steel surface. Polarization curves demonstrated that the examined Bip2 behaves as a mixed type inhibitor. AC impedance results showed that charge transfer resistance increases and the capacitance decreases with increase in the Bip2 concentration. The results obtained from different experimental studies are in good agreement.

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