1-Benzoyl-4-phenyl-3-thiosemicarbazide as Corrosion Inhibitor for Carbon Steel in H_3PO_4 Solution

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Abstract

The inhibition of corrosion of carbon steel in 2 N H_3PO_4 by 1-benzoyl-4-phenyl-3-thiosemicarbazide has been investigated by potentiodynamic polarization and ac impedance techniques. The results showed that inhibitor performance depends on both concentration and temperature. The increasing of temperature reduces the inhibition efficiency. Polarization studied revealed that these compounds behave as mixed-type inhibitors. The inhibition action of 1-benzoyl-4-phenyl-3-thiosemicarbazide was performed via adsorption of this compound on carbon steel surface. The adsorption was spontaneous and followed Langmuir adsorption isotherm. Some thermodynamic parameters are calculated and discussed.

Keywords: Corrosion, Carbon steel, H_3PO_4, 1-benzoyl-4-phenyl-3-thiosemicarbazide

1. Introduction

Phosphoric acid is a medium-strong acid which is widely used in surface treatment of steel such as chemical and electrolytic polishing, chemical coloring, removal of oxide film, phosphating passivating and surface cleaning. But it still shows strong corrosiveness on ferrous or ferrous alloy. Acids are widely used in many industries. Some of the important areas of applications are industrial acid descaling and oil well acidization (Schmitt, 1984). Due to, the aggressiveness of the acids, inhibitors are used to reduce the rate of dissolution of metals. In most inhibitor studies, the formation of donor-acceptor surface complex between free or π-electrons of an inhibitor and vacant d-orbital of the metal was proposed (Abd El Rehim et al, 2002, Selvi et al, 2003 and Agawal et al, 2004). Most of additives used are organic compounds having π bonds in their structures. It is reported that sulphur-containing inhibitors such as sulphooxides (Reihard et al, 1990 and Fignani et al, 1990) sulphides (Tabanelli et al, 1973)and thioureas (Frignani et al., 1987) are found to be efficient in H_2SO_4 environment, whilst nitrogen-containing compounds such as azole derivatives (Nicola et al 1995, Starchak et al, 1994, Chebabe et al., 1995, Quraishi et al, 1995 and Singh et al.,1995) , imidazolin derivatives [Melissa et al., 1992 and Khanin et al., 1994), condensation products of amines with aldehydes [Turbina et al., 1969 Nemchaninova et al, 1974and Komarov et al., 1961), alkyl and aryl amines [Bacskai et al., 1988, perboni et al, 1988, and Jerzykiewicz et al., 1989) perform better in HCl. 4-acetyl pyridine (Vosta et al., 1990), aliphatic amines (Singh et al., 1990), acetylenic alcohols, heterocyclic compounds (Touhami et al., 2000, Lahodny-Sarc et al., 2000, Quraishi et al., 2001, Quraishi et al., 2003, Blin et al., 2004, Khaled, 2003, Amar et al., 2006, Nakai et al., 2004, and Guo et al., 2001) inhibit the corrosion of steel in acid solution by donating the unshared pair of electrons from N atom and form a surface complex. The inhibitive effect of aniline and alkylamine (Luo et al., 1998) p-substituted anilines (Abdel Fattah et al., 1991) is attributed to the interaction of π-electron cloud of aromatic ring on iron and steel surface through vacant d orbital of iron leading to the formation of co-ordination bond between Fe-N. Ortho-substitutesed anilines (Khaled et al., 2003) and polyanilines (Sathiyanarayanan et al, 1992 and Sathiyanarayanan et al., 1994) were also studied.

In this paper the inhibition efficiency of 1-benzoyl-4-phenyl-3-thiosemicarbazide on the corrosion of carbon steel in 2 N H_3PO_4 has been studied by electrochemical methods. Also, the effect of temperature on the corrosion rate was discussed.

2. Experimental details

2.1 Materials

The working electrodes were made of a sheet of carbon steel, which was of commercial specification with composition (weight %): C 0.2; P 0.005; S 0.017; Si 0.017; Mn 0.07 and Fe remainder.

2.2 Solutions

The aggressive solutions were made of 2 N H_3PO_4. Appropriate concentrations of H_3PO_4 were prepared. All chemicals used were of analytical grade. All solutions were prepared from bi-distilled water except the solution of inhibitors in absolute ethanol.

2.3 Inhibitor

The 1-benzoyl-4-phenyl-3-thiosemicarbazide was prepared as before (Rakha et al, 1996). The structure of this 1-benzoyl-4-phenyl-3-thiosemicarbazide was checked by elemental analysis, NMR and IR spectra. This inhibitor is:

Name and Molecular formula	Structure	Molecular Weight
1-benzoyl-4-phenyl-3-thiosemicarbazide $C_{14}H_{13}N_3OS$		271.344
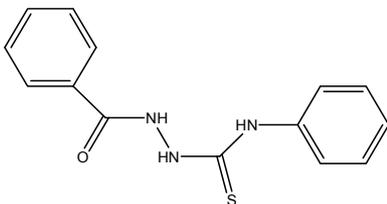
2.4 Electrochemical measurements:

A traditional three-electrode cell was used for electrochemical measurements. The working electrode is carbon steel rods. The rod specimen was embedded in Teflon holder using epoxy resin with an exposed area of 0.7 cm^2. Before each experiment, the electrode was mechanically polished with various grades of emery paper and then ultrasonically cleaned in acetone for 2 min, followed by a rinse in double-distilled water. A platinum sheet electrode was used for the auxiliary electrode, and the reference electrode was a saturated calomel electrode (SCE) with a Luggin capillary. Potentiodynamic polarization studies were carried out on iron in 2 N H_3PO_4 solutions without and with different concentrations of the investigated inhibitors at 30 ^{o}C. All experiments were carried out at 25 ± 1°C. The inhibition efficiency (%η) and the degree of surface coverage (Ө) is calculated from the following equations:

$$\% \, \eta = [\, 1 - (j_{inh.} / j_{corr})] \times 100 \qquad\qquad (1)$$

$$\Theta = [1 - (j_{inh.} / j_{corr})] \qquad\qquad (2)$$

where j_{corr} and j_{inh} are the uninhibited and inhibited corrosion current densities, respectively.

3. Results and discussion

3.1 Potentiodynamic measurement

Figure (1) shows the potentiodynamic polarization curves (E vs. log j) of iron dissolution in 2 N H_3PO_4, in the presence of different concentrations of investigated compound. The corrosion current density ($j_{corr.}$) decreased with increasing the concentration of the investigated 1-benzoyl-4-phenyl-3-thiosemicarbazide which indicates that this compound act as inhibitor and the degree of inhibition depends on the concentration. The polarization curves are shifted in both the positive and the negative directions depending on the magnitude of concentration of the added inhibitor. This indicates that this compound affect dissolution of iron and hydrogen evolution processes. Besides, E_{corr} shifted slightly (< 40 mV) with different inhibitor concentrations, so it can also be deduced that this inhibitor showed a mixed-type character for carbon steel in 2 N H_3PO_4 solution [Ashassi-Sorkhabi et al., 2004), which suppressed both anodic and cathodic reactions by adsorbing on the carbon steel surface and blocking the active sites (Juttner, 1990 and Fawcett et al., 1992).

The corrosion kinetic parameters such as corrosion current density ($j_{corr.}$), corrosion potential (E_{corr}), cathodic Tafel slope (β_c), anodic Tafel slope (β_a), degree of surface coverage (Θ) and percentage inhibition (%η) are recorded in Table (1).

From the results of Table (1), the corrosion potential in the presence and absence of inhibitors is nearly the same; this indicates that the inhibition effect is caused by geometrical blocking of the metal surface electrode by adsorbed inhibiting species (Cao, 1996). Also, the corrosion current densities decrease with increasing of inhibitor concentration.

3.2 Adsorption isotherm

The establishment of adsorption isotherms describes the adsorptive behavior of inhibitor molecules which can provide important ideas to the nature of the metal inhibitor interaction. Adsorption of the organic molecules occurs as the interaction energy between molecule and the metal surface is higher than that between the water molecules and surface (McCafferty , 1979). Basic information dealing with the interaction between the inhibitor molecule and metal surface can be provided by adsorption isotherm (Bentis et al., 2002). In order to find out the adsorption isotherm, attempts were made to fit various isotherms viz., Frumkin, Langmuir and Temkin. However, the best fit was obtained only with Langmuir adsorption isotherm (equation 3) where C/θ was plotted against concentration (C) which resulted with straight lines with slope equal to unity (Figure 2). The equilibrium constant of the adsorption process, K_{ads}, which is related to the standard free energy of adsorption (ΔG^o_{ads}) by [Kliskic et al., 1981 and Abdella et al., 1981).

$$C/\Theta = 1/K + C \qquad\qquad (3)$$

$$K_{ads} = (1/55.5) \exp [-\Delta G^o_{ads} / RT] \qquad\qquad (4)$$

where R is the universal gas constant and T is the absolute temperature, C is inhibitor concentration and K_{ads} is equilibrium constant of adsorption. Also, it is found that the kinetic– thermodynamic model of El-Awady et al (El-Awady et al., 1985)

$$\log (\Theta/1-\Theta) = \log K^\backslash + y \log C \qquad\qquad (5)$$

is valid to operate the present adsorption data. $K_{ads} = K^\backslash_{ads}(1/y)$, $K^\backslash_{ads}$ is constant, and 1/y is the number of the surface active sites occupied by one inhibitor molecule. The negative values of ΔG^o_{ads} (Table 2) obtained here indicate that the adsorption process of these compounds on the carbon steel surface is spontaneous one. The higher values of K_{ads} suggested strong interaction of the inhibitor on the carbon steel surface (Oguzie, 2007). The increasing of K_{ads} reflects the increasing adsorption capability, due to structural formation on the metal surface (Kliskic,., 1997).

3.3 Kinetic-thermodynamic corrosion parameters

The effect of temperature (25- 55 ^{o}C) on the corrosion rate of carbon steel in 2 N H_3PO_4 at different concentrations of inhibitors was studied using potentiodynamic polarization measurement (Fig. 4). Plots of log k (corrosion rate) against 1/T (Fig. 5), for iron dissolution in 2 N H_3PO_4 at different concentrations of investigated inhibitor, give straight lines. The value of the slope obtained at different temperatures permit the calculation of Arrhenius activation energy (E_a^*). The activation energy values obtained from this Figure are tabulated in Table 3. Activation parameters for corrosion of iron in 2 N H_3PO_4 were calculated from Arrhenius–type plot:

$$k = A \exp (-E_a^*/ RT) \qquad\qquad (4)$$

and transition state type equation:

$$k = RT/ Nh \exp (\Delta S^*/ R) \exp (-\Delta H^*/RT) \qquad\qquad (5)$$

The almost similar values of ΔE_a^* suggested that the inhibitors are similar in the mechanism action and the order of the efficiency many be related to the preexponential factor A in equation (4). This is further related to concentration, steric effect, and metal surface characters.

The relationship between log k/T vs. 1/T gives straight line (Fig. 6), from its slope, ΔH^* can be calculated and from its intercept ΔS^* can be also calculated. The presence of investigated inhibitor increases the activation energy of iron indicating strong adsorption of the inhibitor molecules on the metal surface. The presence of this additive induce energy barrier for the corrosion reaction and this barrier increases with increasing the additive concentration. The value of ΔH^* are reported in Table (3) and is positive. The positive sign of the enthalpy reflects the endothermic nature of carbon steel dissolution process (Quartarone et al., 1994). Also, the value of entropy of activation is negative. The negative value of entropy imply that the activated complex in the rate

determining step represents an association rather than dissociation step, meaning that a decrease in disordering takes place on going from reactant to activated complex (Abdallah et al., 2006).

A decrease in inhibition efficiency with rise in temperature, suggests a possible desorption of some adsorbed inhibitor molecules from the metal surface at higher temperatures. Such behavior indicates that inhibitor molecules were physically adsorbed on the metal surface (Fouda et al., 1986) .

3.4 Electrochemical impedance spectroscopy (EIS)

The corrosion of carbon steel in 2 N H_3PO_4 in the presence of 1-benzoyl-4-phenyl-3-thiosemicarbazide was investigated by EIS method at 25 $^{\circ}$C after 20 min immersion. Nyquist and Bode plots in the absence and presence of investigated compound are presented in Figs. 7 and 8. It is apparent that all Nyquist plots show a single capacitive loop, both in uninhibited and inhibited solutions. The impedance data of carbon steel in 2 N H_3PO_4 are analyzed in terms of an equivalent circuit model which includes the solution resistance R_s or R_Ω and the double layer capacitance C_{dl} which is placed in parallel to the charge transfer resistance R_{ct} (Sekine et al., 1992)] due to the charge transfer reaction. For the Nyquist plots it's obvious that low frequency data are on the right side of the plot and higher frequencies are on the left. This is true for EIS data where impedance usually falls as frequency rises (this is not true of all circuits). The capacity of double layer (C_{dl}) can be calculated from the following equation:

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

where f_{max} is maximum frequency. The parameters obtained from impedance measurements are given in Table (4). It can see from Table (4) that the values of charge transfer resistance increase with inhibitor concentration (Larabi et al., 2006). In the case of impedance studies, % IE increases with inhibitor concentration in the presence of investigated inhibitor. The impedance study confirms the inhibiting characters of this compound obtained with potentiodynamic polarization and ac impedance methods. It is also noted that the (C_{dl}) values tend to decrease when the concentration of this compound increases. This decrease in (C_{dl}), which can result from a decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer, suggests that this compound molecules function by adsorption at the metal/solution interface (Lagrenee et al., 2002).

The inhibiting effect of this compound can be attributed to the parallel adsorption at the metal solution interface. The parallel adsorption is owing to the presence of one or more active center for adsorption. The chemisorptions take place by the formation of a chemical bond between the metal and the adsorbed molecule. Chemisorption involves charge or charge transfer from inhibitor molecule to the metal surface forming co-ordinate type bond (Bellucci et al., 1991).

3.5 Chemical structure and corrosion inhibition

The inhibition efficiency of the investigated compounds depends on many factors, which include the number of adsorption active centers in the molecule and their charge density, molecular size, mode of adsorptions, heat of hydrogenation and formation of metallic complexes.

In aqueous acidic solutions, this inhibitor exists either as neutral molecule or as protonated molecule (cation). This inhibitor may adsorb on metal/acid solution interface (Schweinsberg et al., 1988) by one and/or more of the following ways: (i) electrostatic attraction between charged molecules and charged metal, (ii) interaction of unshared electron pairs in the molecule with the metal, (iii) interaction of π electrons with the metal and (iv) a combination of the three before.

In general, two modes of adsorption are considered on the metal surface in acid media. In one mode, the neutral molecules may be adsorbed on the surface of steel via chemisorptions mechanism, involving the displacement of water molecules from the carbon steel surface and the sharing electrons between the hetero-atoms and carbon steel. The inhibitor molecule can also adsorb on the steel surface on the basis of donor-acceptor interactions between π-electrons of the aromatic ring and vacant d-orbital of surface iron atom. In the second mode, since it is well known that the carbon steel surface bears positive charge in acid solution (Mu et al., 1996), so it is difficult for the protonated molecules to approach the positively charged nickel surface due to the electrostatic repulsion. $PO4^{-3}$ ions could bring excess negative charges in the vicinity of the interface and favor more adsorption of the positively charged inhibitor molecules, the protonated inhibitor adsorb via electrostatic interactions between the positively charged molecules and negatively charged metal surface. Thus there is a synergism between adsorbed PO^{-3} ions and protonated inhibitor. Thus we can conclude that inhibition of carbon steel corrosion in 2 N H_3PO_4 is mainly due to electrostatic interaction. The decrease in inhibition efficiency with rise in temperature supports electrostatic interaction.

The high percentage inhibition efficiency of this compound may be due to the presence of three nitrogen, one oxygen and one sulphur atoms in the molecule.

4. Conclusions

1) This additive is found to perform well as a corrosion inhibitor in 2 N H_3PO_4 solution and the inhibiting efficiency reach to about 95 % at 21 x 10^{-5} M.

2) This investigated compound is found to act as mixed–type inhibitor.

3) The corrosion process is inhibited by the adsorption of these inhibitor molecules on carbon steel surface, following Langmuir's adsorption isotherm. The inhibition efficiency increased with increase in inhibitor concentration and decreased with increase in temperature.

4) Electrochemical studies gave similar results.

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Table 1. Potentiodynamic polarization parameters for corrosion of carbon steel in 2 N H_3PO_4 in the absence and presence of different concentrations of investigated compound at 25 ^{o}C

Conc. $\times 10^5$ M	$-E_{corr}$ mVvs.SCE	i_{corr} $\mu A\ cm^{-2}$	β_a mV dec^{-1}	β_c mV dec^{-1}	$R_p \times 10^2$ $\Omega\ cm^2$	θ	% η
Blank	481	814.9	350	435	1.035	0	0
1	474	275.4	266	343	2.366	0.662	66.2
5	481	118.3	249	310	5.072	0.855	85.5
9	475	79.34	235	287	7.082	0.903	90.3
13	468	66.76	257	293	8.928	0.918	91.8
17	470	47.58	238	272	11.62	0.942	94.2
21	470	44.21	230	269	12.19	0.946	94.6

Table 2. Equilibrium constant (K), adsorption free energy (ΔG°_{ads}) and number of active sites (1/y) for the adsorption of the inhibitor (C) on C-steel in 2 N H_3PO_4 at 25 ^{o}C

Langmuir isotherm				Kinetic model	
Intercept x 10^{-5}	K_{ads} x 10^{-4} M^{-1}	$-\Delta G^\circ_{ads}$ kJ mol^{-1}	1/y	K_{ads}x 10^{-4} M^{-1}	$-\Delta G^\circ_{ads}$ kJ mol^{-1}
0.62	16.13	39.67	1.384	24.5	40.7

Table 3. Activation parameters for dissolution of carbon steel in 2 N $H_3 PO_4$ in the presence of 21×10^{-5} M of inhibitor (C)

Inhibitor	Ea*, kJ mol^{-1}	ΔH^*, kJ mol^{-1}	$-\Delta S^*$, J mol^{-1}k^{-1}
1-benzoyl-4-phenyl-3-thiosemicarbazide	68.7	66.1	107.2

Table 4. Electrochemical impedance spectroscopy (EIS) parameters for carbon steel in 2 N H_3PO_4 in the absence and presence of different concentrations of inhibitor (C) at 25 °C.

Conc.x10^{-5} M	C_{dl}, $\mu F\ cm^{-2}$	R_{ct}, $\Omega\ cm^2$	θ	% η
0	81.27	19.91	0	0
5	76.78	138.6	0.856	85.6
13	38.25	251.2	0.921	92.1
17	28.54	698.7	0.972	97.2
21	26.96	1027	0.981	98.1
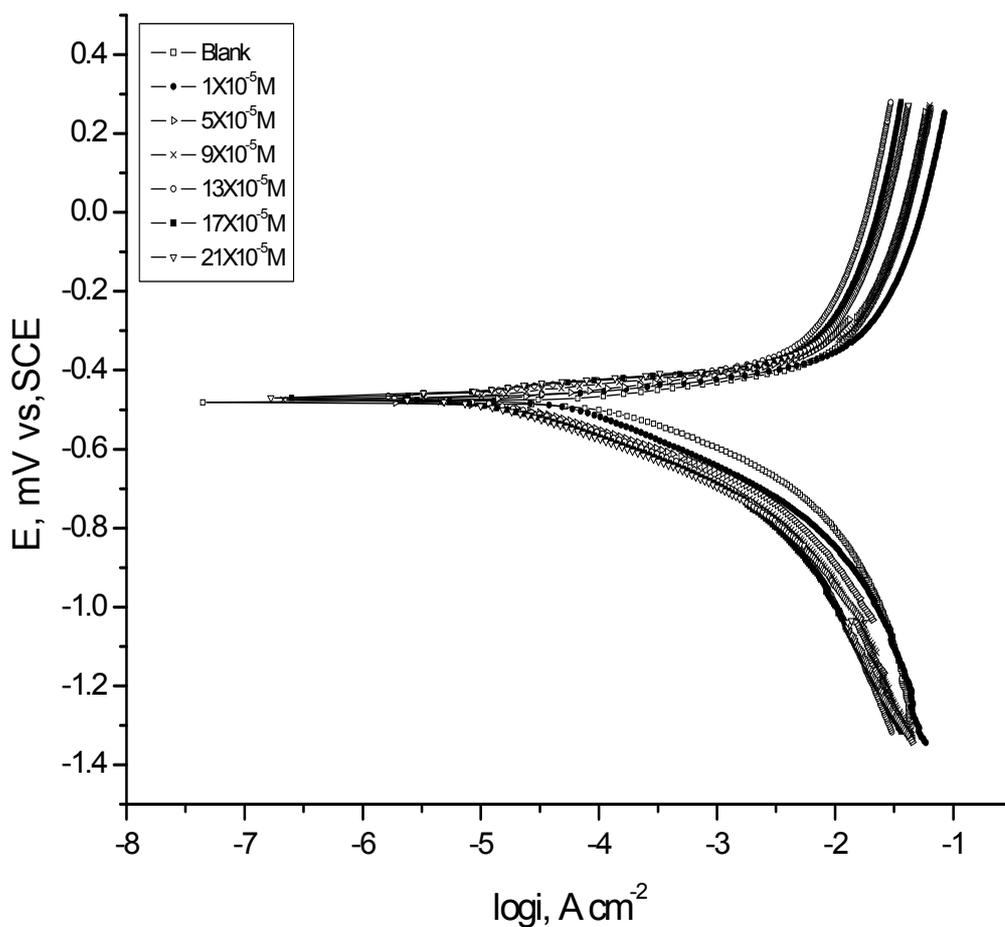
Figure 1. Polarization curves for the dissolution of C-steel in 2 N H_3PO_4 in the absence and presence of different concentrations of investigated compound at 25 °C
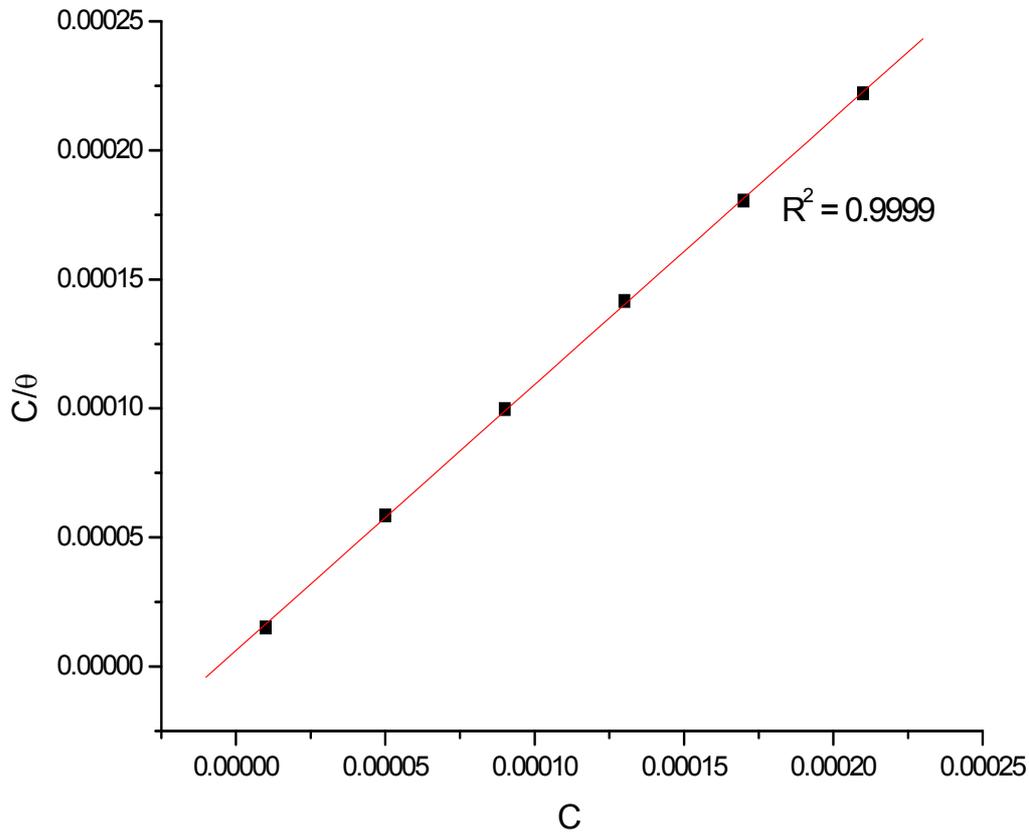
Figure 2. Langmuir adsorption isotherm for carbon steel in 2 N H_3PO_4 in the presence of different concentrations of investigated compound at 25 ^{o}C
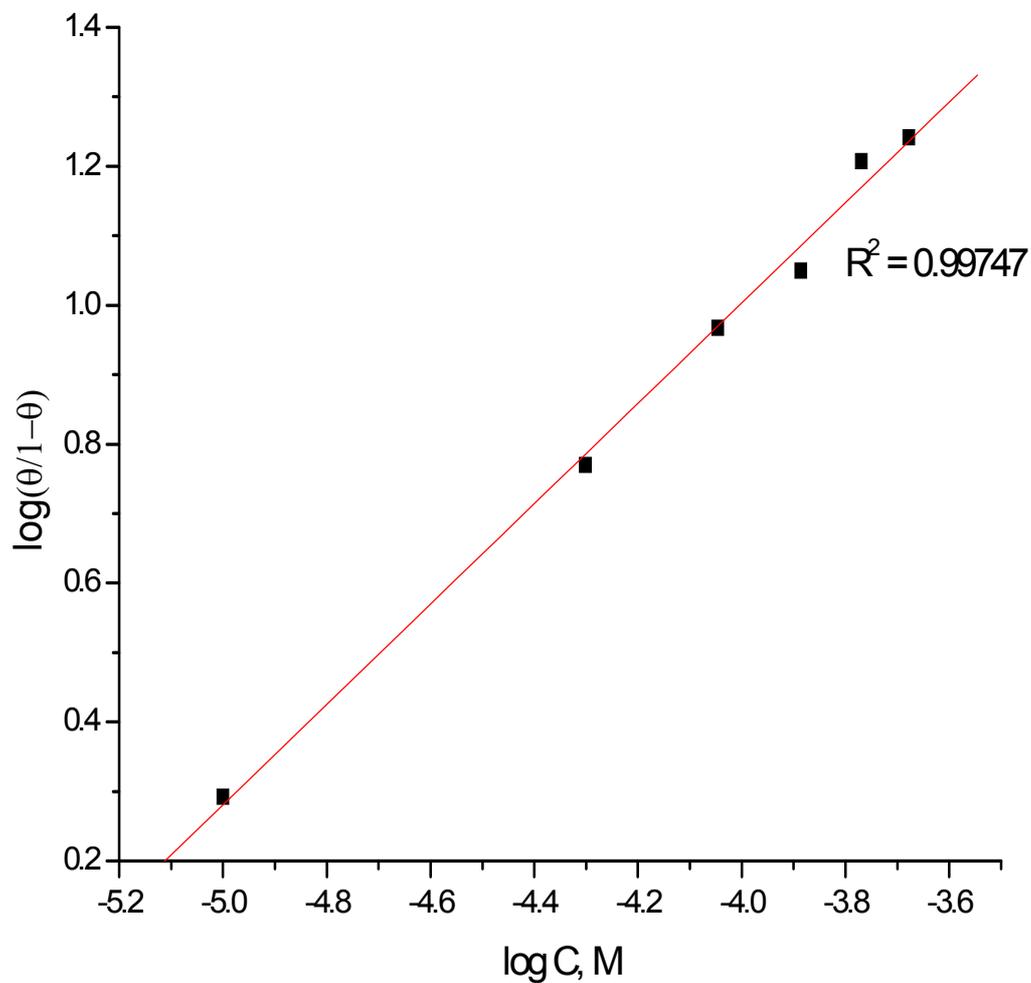
Figure 3. El- Awady model plotted as log ($\theta/1-\theta$) vs. log C of investigated compound for corrosion of carbon steel in 2 N H_3PO_4 solutions at 25 °C
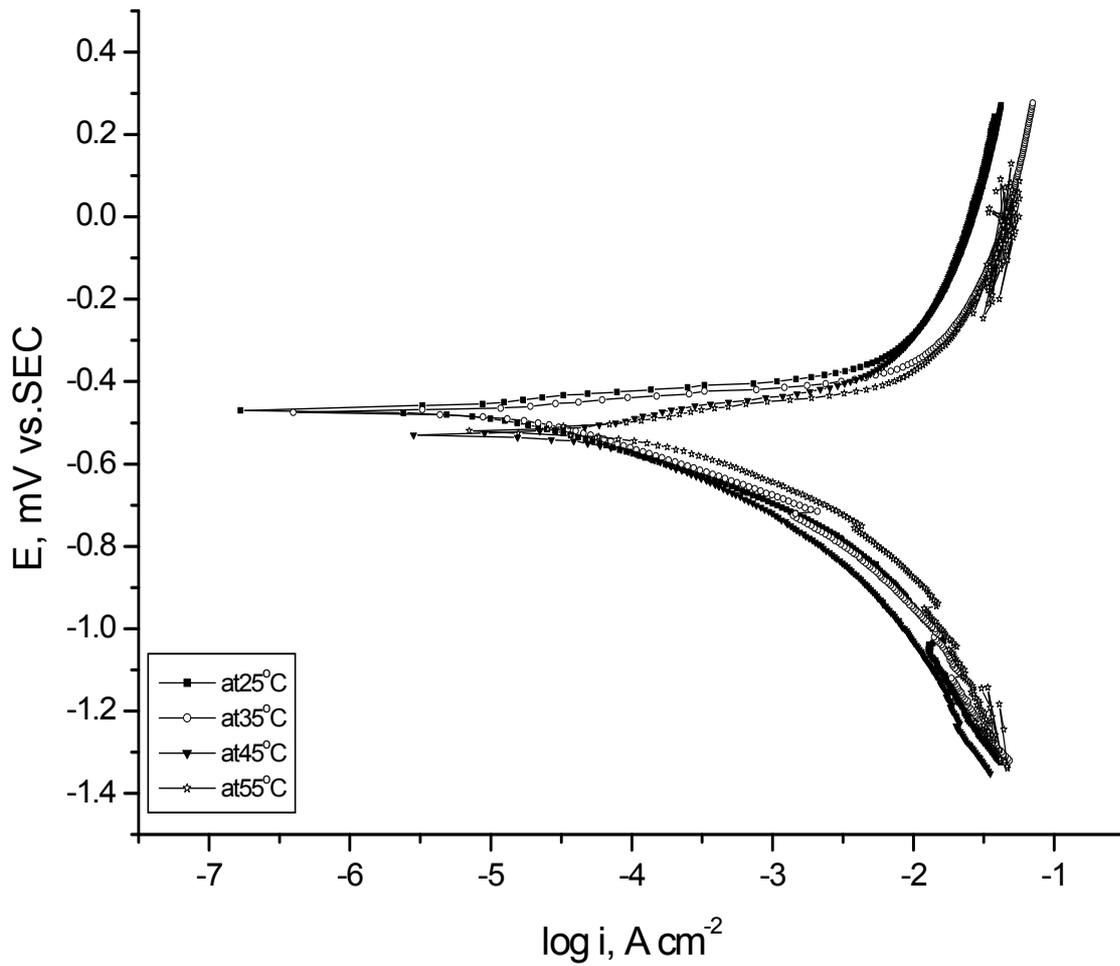
Figure 4. Polarization curves for dissolution of carbon steel in 2 N H_3PO_4 in the presence of 21×10^{-5} M of investigated compound at different temperatures.
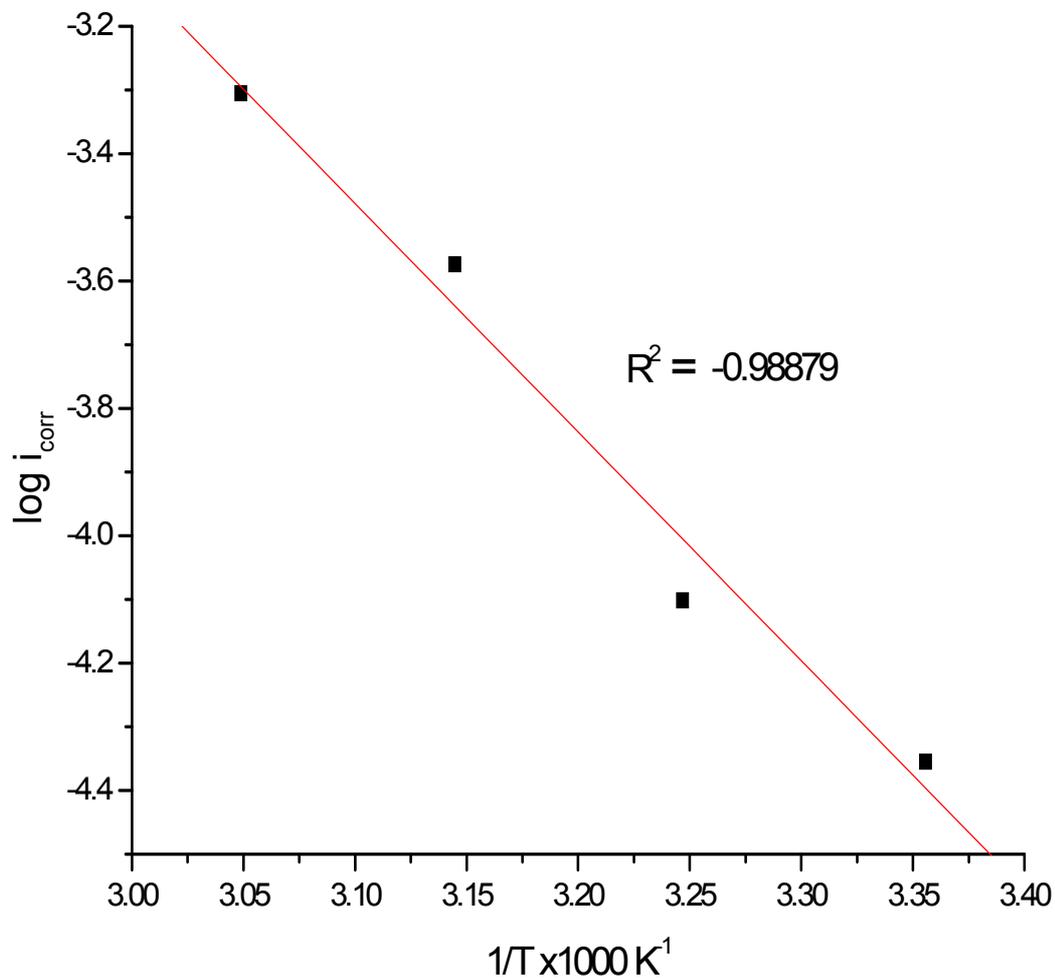
Figure 5. Arrhenius plot (log i_{corr}-1/T) for carbon steel dissolution in 2 N H_3PO_4 in the presence of 21×10^{-5} M inhibitor (C)
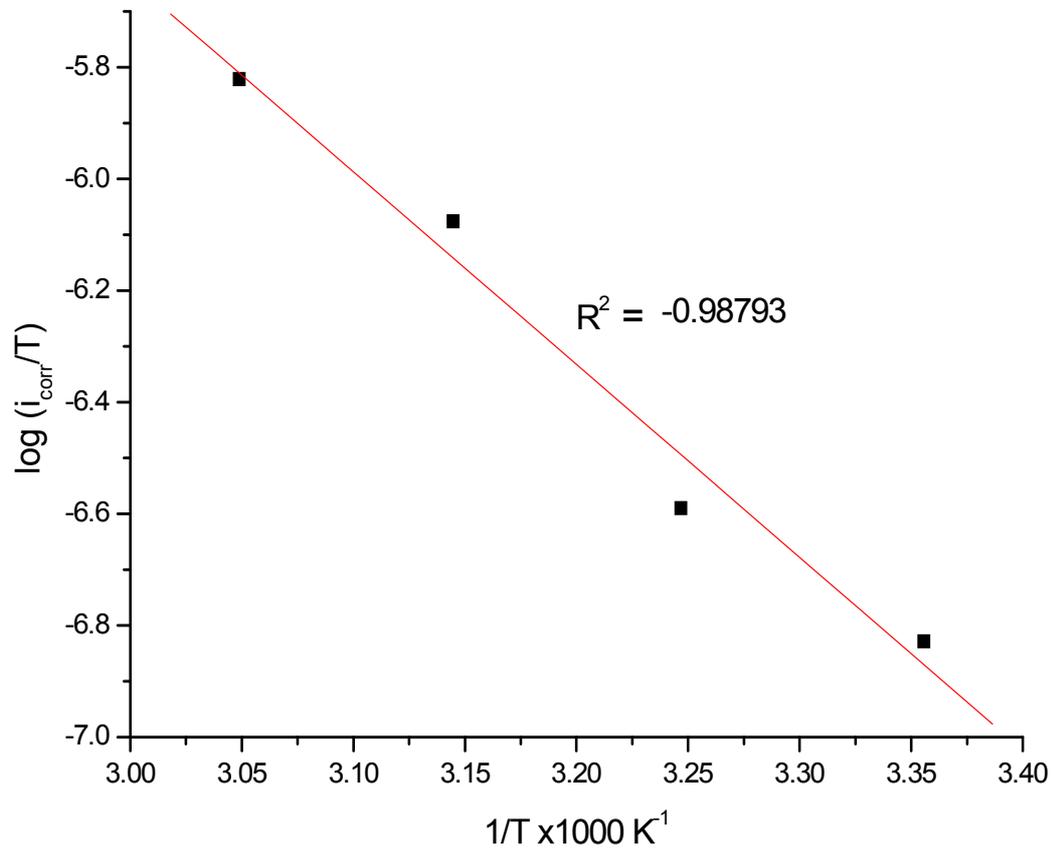
Figure 6. Arrhenius plot (log i_{corr} / T vs. 1/T) for carbon steel dissolution in 2 N H_3PO_4 in the presence of 21×10^{-5} M inhibitor (C) .
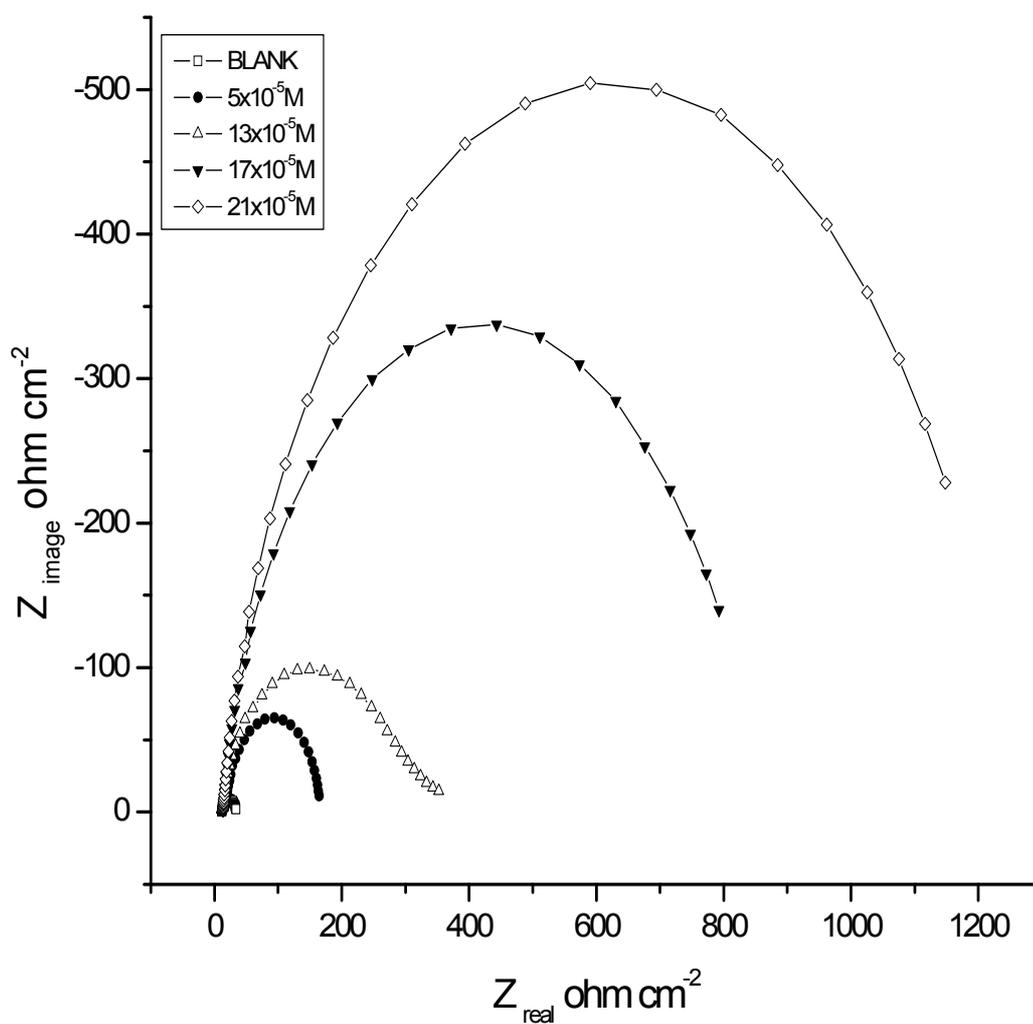
Figure 7. The Nyquist plots for carbon steel dissolution in 2 N H_3PO_4 in the absence and presence of different concentrations of investigated compound at 25 ^{o}C
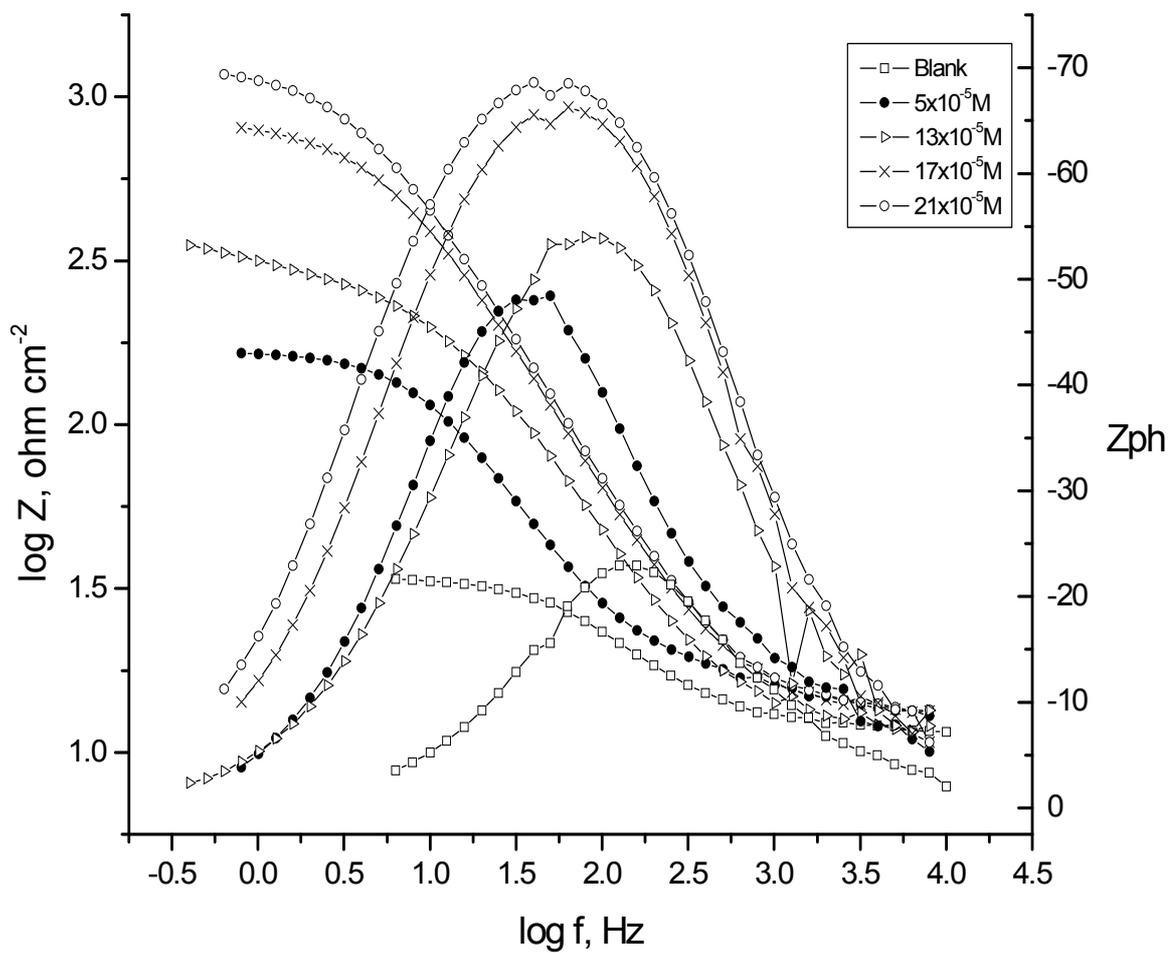
Figure 8. The Bode plots for carbon steel dissolution in 2 N H_3PO_4 in the absence and presence of different concentrations of inhibitor (C) at 25 ^{o}C