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6-phenylpyridazin-3(2H)one as New Corrosion Inhibitor for C38 Steel in 1 M HCl.

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The corrosion behavior of C38 steel in HCl solutions and its inhibition by 6-phenylpyridazin-3(2H)one (**PPO**) has been studied in different temperature using polarization and electrochemical impedance spectroscopy (EIS) techniques as well as weight loss measurements. The 6-phenylpyridazin-3(2H)one has shown good inhibitive properties and acts as mixed inhibitor as observed in polarization method. The *EIS* results confirmed the efficiency of tested inhibitor and showed that the charge transfer resistance increase with the rise in the concentration following the same trend of the inhibition efficiency. The effect of the temperature, Langmuir adsorption isotherm and their parameters are discussed to examine the mechanism of the interactions between metal surface and tested compound. A chemisorption mechanism is proposed.

Keywords: C38 steel, corrosion inhibition, 6-phenylpyridazin-3(2H)one, Langmuir.

1. INTRODUCTION

Hydrochloric acid is one of the popularly solutions used in industry in different applications such as acid pickling and oil well cleaning. These industrial process leads corrosive environments which rise the degradation of the carbon steel used in most area of industry [1–6]. This unfavorable problem is one of the interesting research fields in academic and research and development (R&D) teams [7-8]. Different methods are developed by the researchers as well as engineering such as coating and material selection…*etc.* but, due to the economic considerations and availability, the protection of the metals using organic chemical compounds as corrosion inhibitors became one of the convenient methods used in this case. The presence of the most electronegative atoms such as nitrogen, oxygen and sulfur as well as the electron cloud in the aromatic ring such as benzene, pyridine and pyridazine are the important characteristic associated to the efficient corrosion inhibitors [9, 10]. The chemical compounds with these properties can easily adsorbed on the metal surfaces resulting an important decrease in the corrosion rate by blocking the active sites. The most reported types of the interactions during the adsorption of the inhibitors are the electrostatic interactions between charged metallic surface and charged species in the bulk solution, and chemical interactions *via* charge transfer from organic molecules to metallic surface, others compounds can be adsorbed through chemical and electrostatic interactions [11–17].

In this investigation, the inhibition performance of 6-phenylpyridazin-3(2H)one named in the abbreviation form **PPO** as inhibitor of CS corrosion in acidic medium is examined by different techniques such as weight loss as baseline method and electrochemical techniques (*PDP* and *EIS*). The structure of the 6-phenylpyridazin-3(2H)one is shown in Fig.1**.**

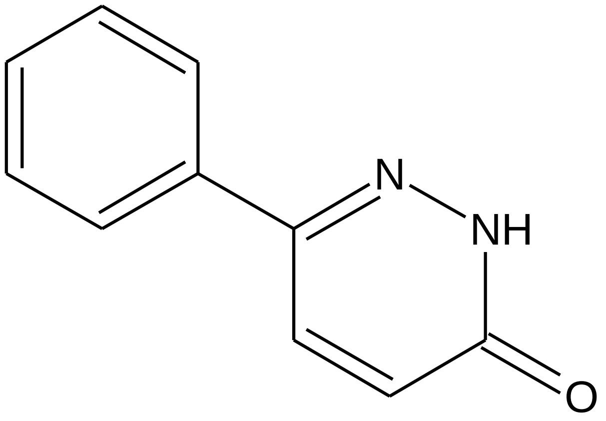

Figure 1. Molecular structure of 6-phenylpyridazin-3(2H)one (**PPO**)

2. MATERIALS AND METHODS

2.1. Electrochemical and weight loss tests

The investigation of the effect of the 6-phenylpyridazin-3(2H)one (**PPO**) on the C38 steel corrosion were carried out using electrochemical techniques (*EIS* and weight loss (*WL*) at 298 K), while the *PDP* experiments performed at 298 to 328 K. More details of experiments and used materials can be obtained in our previous works such as [18–20].

In this study, the *PDP* measurements were obtained in the interval of −800 mV to −200 mV/SCE with a scan rate 1 mV/s. The *IE* (%) for PDP measurements were calculated with the help of the corrosion current densities in blank solution I_{corr} and with 6-phenylpyridazin-3(2H)one (**PPO**) concentrations as follows:

$$EI_{I_{corr}} = \frac{I^{\circ}_{corr} - I_{corr}}{I^{\circ}_{corr}} \times 100 \qquad (1)$$

where I_{corr} and I°_{corr} are the without and with 6-phenylpyridazin-3(2H)one (**PPO**) concentrations.

EIS study were performed with a small alternating voltage perturbation (10 mV) at frequencies between 100 kHz and 10 mHz at open circuit potential at 298 K. The inhibition efficiencies (%) in the case of the EIS measurements were calculated using charge transfer resistance values in blank solution R_t and with 6-phenylpyridazin-3(2H)one (**PPO**) concentrations R^0_t as follows:

$$EI_{Rt}(\%) = \frac{R_t - R^0_t}{R_t} \times 100 \qquad (2)$$

2.2. Materials and solutions

The aggressive medium (1M HCl) were prepared by diluting a reagent of analytical grade HCl 37% (from Sigma-Aldrich) with double-distilled water. The compositions of the CS used in this study are 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 balance Fe. The 6-phenylpyridazin-3(2H)one (**PPO**) used as corrosion inhibitor is presented in Fig. 1, the molecule of this compound contain heteroatoms and aromatic ranges which can rise their ability as effective corrosion inhibitor for CS in 1 M HCl solution. The concentration of **PPO** employed in this investigation was 10^{-6} to 10^{-2} M in 1 M HCl.

3. RESULTS AND DISCUSSION

3.1. Effect of concentration

3.1.1. Polarisation method

The polarisation curves of CS in acidic medium alone and containing various concentrations of 6-phenylpyridazin-3(2H)one (10^{-6} to 10^{-2} M) are graphically presented in Fig.2. It is observed that both reactions *i.e.*, anodic and cathodic, were inhibited in the presence of tested compound which is retarded the hydrogen evolution reaction and metal dissolution after addition of 6-phenylpyridazin-3(2H)one inhibitor.

The parameters of the polarization results (E_{corr}, I_{corr} and βc) for uninhibited and inhibited solutions as well as calculated inhibition efficiency for the corrosion process of CS in hydrochloric solutions are collected in Table 2.

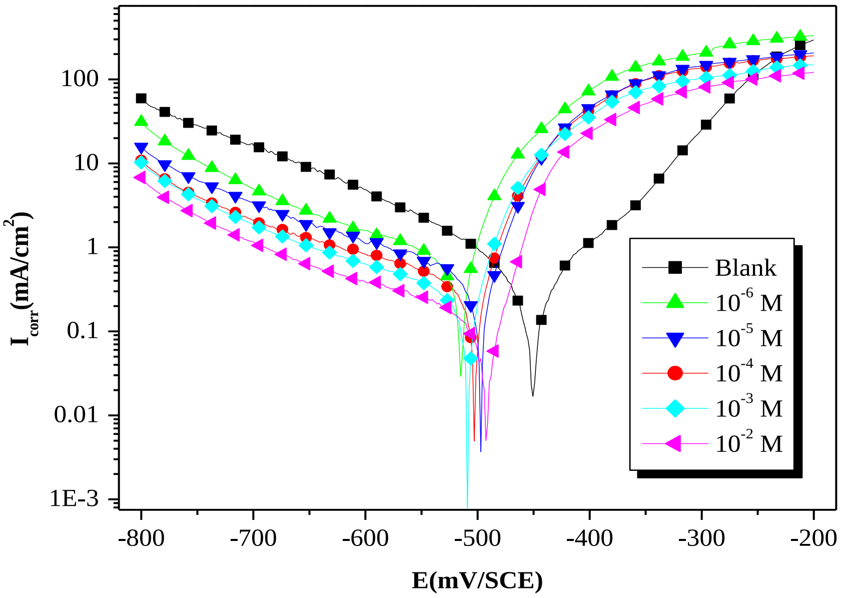

Figure 2. Polarisation plots of CS in 1 M HCl containing **PPO** at 298 K.

Table 1. The parameters of CS corrosion derived from polarization experiments without and with tested **PPO**.

Concentration (M)	E_{corr} (mV/SCE)	I_{corr} (mA/cm^2)	$-\beta_c$ (mV/dec)	IE_{Icorr} (%)
Blank	-450	594	186	----
10^{-6}	-508	374	146	**37.03**
10^{-5}	-504	257	168	**56.73**
10^{-4}	-501	202	165	**65.99**
10^{-3}	-511	155	147	**73.90**
10^{-2}	-491	107	148	**81.98**

The corrosion current density (I_{corr}) decreased considerably from 594 (in blank solution) to 107 mA/cm^2 (at 10^{-2} M of **PPO**). This concentration effect leads an increase of the inhibition efficiency to reaches 81.98% at 10^{-2} M. On the other hand, it is noticed that a small displacement of the corrosion potential as well as insignificant changes in cathodic Tafel slope in corrosion process is mainly an indication that the compound is mixed type inhibitor, *i.e.*, shifts cathodic and anodic reactions without changing the mechanism of both reactions [21, 22]. In the present investigation, the corrosion inhibition process is controlled by all these characteristics. In order to comparing these results, Bouklah et al., [23] investigated the corrosion inhibition of the MS using two pyridazine derivatives, noted P1 and P2, the authors reported that the addition of these compounds in corrosive medium (0.5 M of H_2SO_4) solutions decrease the corrosion rate and increase the inhibition efficiency to reach maximum value (100%) in presence of P1 at 5.10^{-4}. Similar results are presented in previous study [24] using pyridazine derivatives in order to find any effect of the molecular structure of tested inhibitors and its inhibitive properties for iron corrosion inhibition in 1M HCl medium.

3.1.2. Electrochemical impedance spectroscopy measurement

The *EIS* experiments for CS corrosion in 1M HCl were recorded after 0.5h of immersion in dependence on the concentration of 6-phenylpyridazin-3(2H)one. The Nyquist diagrams for uninhibited and inhibited solutions are graphically presented in Fig.3.

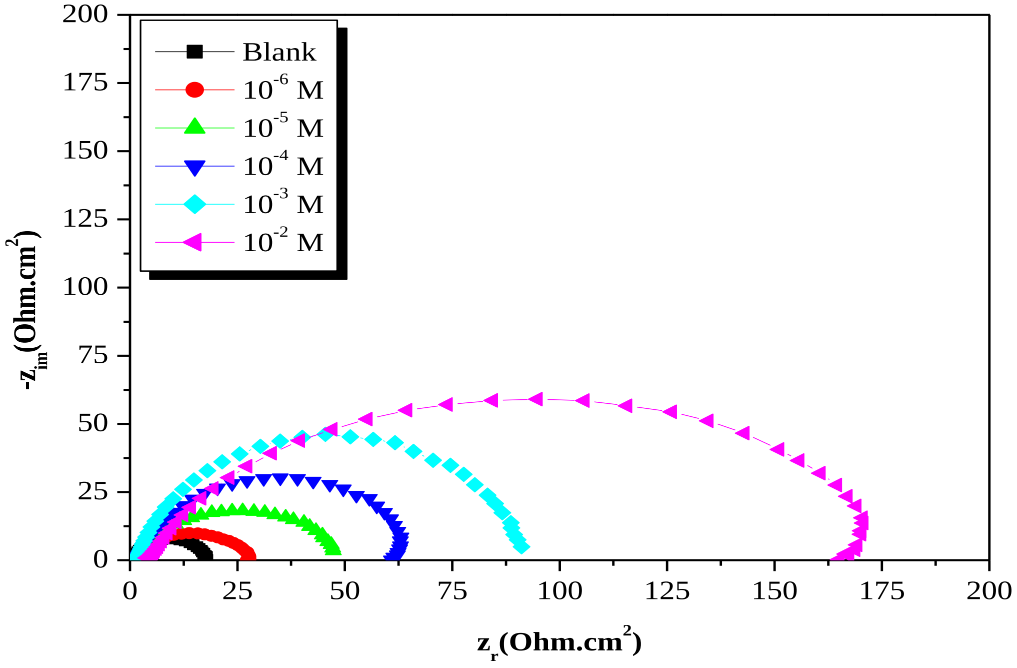

Figure 3. Nyquist presentation of the *EIS* results for CS with and without **PPO**.

From the Fig. 3, the diameter of observed one single semicircle found to depend on the concentration of 6-phenylpyridazin-3(2H)one. It increases with the increase of the concentration of 6-phenylpyridazin-3(2H)one. Such appearance may indicate that the CS corrosion process is mainly controlled by the charge transfer and that the mechanism of the CS dissolution does not change after addition of 6-phenylpyridazin-3(2H)one (**PPO)** [25]. The Nyquist diagrams recorded are in not perfect form which may be a result of the frequency dispersion of interfacial impedance mainly associated to the inhomogeneity and/or others interfacial phenomena [25-29]. An appropriate equivalent circuit consists of solution resistance R_s, CPE, constant phase element and charge transfer resistance R_t (Fig. 4) is employed to simulate the experimental *EIS* results, an excellent analysis of *EIS* results using *Zview* software is obtained with this equivalent model from which the parameters of the *EIS* experiments presented in Table 2 are estimated [30-31]. All concentrations present good conformity as presented in Fig. 5 as an example of 10^{-5} M of **PPO.**

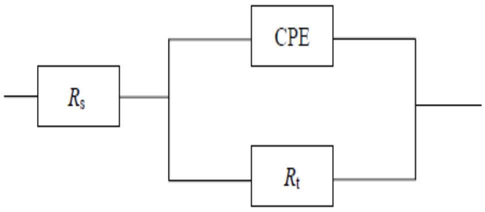

Figure 4. Equivalent circuit model

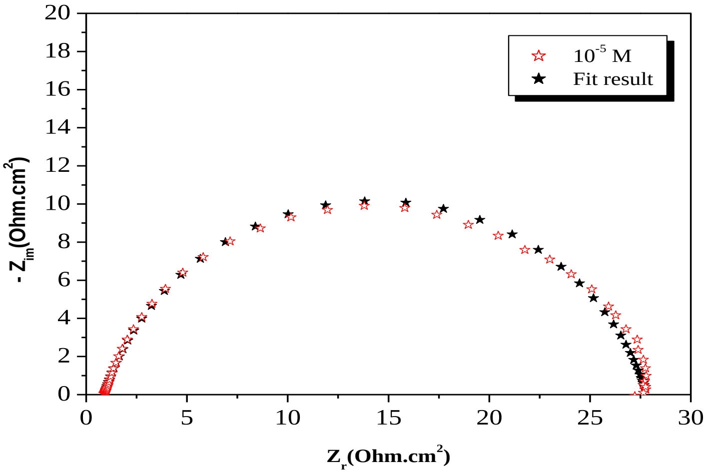

Figure 5. An example of the best fitting of the *EIS* results for 10^{-5} M of **PPO**.

In the same case, the parameters estimated such as Q_{dl} , n and f_{max} (frequency at which imaginary value reaches a maximum on the Nyquist plot) are used to calculate the double layer capacitance using following relation:

$$C_{dl} = Q_{dl} \mathrm{x} (2\pi f_{max})^{n-1} \qquad (3)$$

Table 2. The *EIS* data for corrosion of CS in hydrochloric acid medium with different contents of **PPO**

Concentration (M)	R_t (Ω.cm^2)	n	Q ($s^n/\Omega.cm^2$)	C_{dl} ($\mu F/cm^2$)	IE_{Rt} (%)
Blank	18.00	0.88	21.02×10^{-5}	98.27	----
10^{-6}	26.96	0.78	6.64×10^{-5}	118.05	**33.23**
10^{-5}	49.86	0.75	119×10^{-4}	97.49	**63.89**
10^{-4}	63.72	0.71	2.10×10^{-4}	41.95	**71.75**
10^{-3}	91.50	0.76	2.84×10^{-4}	22.94	**80.32**
10^{-2}	163.24	0.78	2.81×10^{-4}	11.14	**88.97**

From results presented in Table 2, we can be noted that the values of C_{dl} are directly proportional to the 6-phenylpyridazin-3(2H)one concentration, more increase in the concentration of tested inhibitor leads more decrease in the C_{dl} which is referred to the adsorption of the 6-phenylpyridazin-3(2H)one molecules resulting an increase in the film thickness formed on the surface of CS and thus decreases the degradation of the metal as well as the retardation of hydrogen evolution [27]. In the same case, the increase of the concentration of 6-phenylpyridazin-3(2H)one leads to the increase in both, R_t and *EI*%.

As comparison with the published findings, Ghazoui et al., [32] studied the inhibition mechanism of the corrosion of steel in acid solution (1M of HCl), the results presented in the electrochemical impedance section showed that the addition of the two pyridazine derivatives, noted GP2 and GP3, increase the charge transfer resistance from 33.2 (in blank test) to 203.7 and 273.2 Ω cm^2 for GP2 and GP3 respectively at 10^{-3} M, and decreases considerably the C_{dl}. The authors associated these results to the increase of molecules adsorption of two pyridazine derivatives with a rise of their concentrations which can leads a high protection of the steel against deterioration in hydrochloric solution. Others authors [33,34] applied different experimental techniques for investing the inhibition of corrosion of various types of steels in 1M of HCl using pyridazine derivatives, similar conclusions are reported, *i.e.*, the pyridazine derivatives reduce the corrosion effect of the aggressive medium leads an important protection of the metals. These works further support the use of these compounds to protect metals against dissolution.

3.1.3. Weight loss tests

Table 3. The *WL* parameters of CS in acidic medium with presence of 6-phenylpyridazin-3(2H)one

Conc. (M)	W_{corr} (mg. cm^{-2})	**IE_w (%)**
Blank	1.26	**----**
10^{-6}	0.7485	**40.12**
10^{-5}	0.5339	**57.62**
10^{-4}	0.2891	**77.05**
10^{-3}	0.2348	**81.36**
10^{-2}	0.1354	**89.25**

It’s very reported that the inhibitive action, *i.e.*, the adsorption of the chemical compounds used as corrosion inhibitors on the surface of the metals is mainly due to the presence of the reactive sites such as heteroatoms, π-bonds and aromatic rings. These characteristics are very presented in our pyridazine derivative which lets us performing the weight loss tests as baseline method in order to obtained basic information about the efficiency of tested compound. Table 3 collected obtained results such as the inhibition efficiency and corrosion rate before and after 6h of immersion in 1 M HCl for the corrosion inhibition of CS, the *EI*% values are estimated after the evaluation of the corrosion rate in blank medium (W_{corr}) and with presence of 6-phenylpyridazin-3(2H)one (W'_{corr}) using following relation:

$$P\ \% = \frac{W_{corr} - W'_{corr}}{W_{corr}} \times 100 \qquad (4)$$

In this section, the weight loss tests further confirm the dependence of the concentration to the efficiency of tested inhibitor, more increase in the values of the *IE*% is clearly observed when inhibitor concentration increases while the corrosion rate considerably decreases from 1.26 (in blank test) to 0.1354 mg. cm^{-2} at 10^{-2} of **PPO** [33].

3.2. Adsorption isotherm

In order to provide significant information about the adsorption of the chemical compounds used as inhibitors, the adsorption isotherms are very popularly and widely used for the investigation of the interactions types and to provide basic insights about the adsorption modes (Chemical and physical or both). It’s very reported that the adsorption of an organic compound is directly related to the surface coverage ($\theta = EI/100$) from which different adsorption isotherms models such as Langmuir, Frumkin and Temkin are tested. In the present study, according to the correlation coefficient and the slope, the Langmuir model found (Eq. 5) to be the best descriptor for the adsorption process as graphically presented in Fig. 6:

$$\frac{C}{\theta} = \frac{1}{K} + C \qquad (5)$$

with

$$K = \frac{1}{55{,}5} \exp\left(-\frac{\Delta G^0{}_{ads}}{RT}\right) \qquad (6)$$

where C is the Concentration of 6-phenylpyridazin-3(2H)one and K is the adsorption equilibrium constant of the adsorption process.

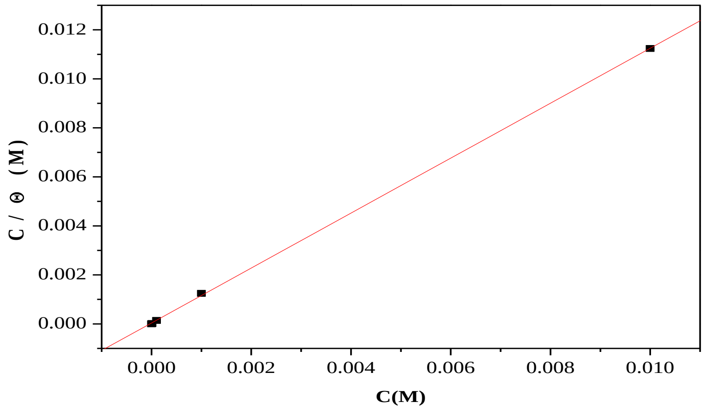

Figure 6. Langmuir plot for CS in 1M HCl with 6-phenylpyridazin-3(2H)one concentrations at 298K.

As reported, the value of equilibrium adsorption constant and ΔG_{ads} related to the adsorption process are the main adsorption parameters in the corrosion inhibition studies. In this case, the large value of K_{ads} as obtained in this study (2.68 x 10^4 M^{-1}) indicates the strong adsorption of the tested **PPO** and the stability of formed protective film. In accordance, the largest negative value of ΔG_{ads}, i.e., -35.19 kJ/mol for 6-phenylpyridazin-3(2H)one, is an indication of the spontaneous chemical interactions between adsorbed 6-phenylpyridazin-3(2H)one and CS surface, the values of the *EI*% obtained in the temperature effect section mainly support the chemical interactions[35-36]. It's reported that the most compounds used as corrosion inhibitors contain various reactive centers such as aromatic ranges and heteroatoms especially the nitrogen, oxygen and sulfur atoms. In this case, the tested compound containing two nitrogen atoms and oxygen atom as well as π-bonds and phenyl group which further rise the adsorption ability of this compound on studied surface of metal. In the case of pyridazine, many researchers found that this type of inhibitors are strongly adsorbed on the surface of steels. The *EI*% of the pyridazine derivative examined by Zarrok et al., [33] namely, 5-(2 chlorobenzyl)-2,6-dimethylpyridazin-3-one decreased with the increase of the temperature but found to be strongly adsorbed in standard conditions. Same observations can be concluded in the work of Ghazoui et al. [32]

3.3. Effect of temperature:

In order to evaluate the temperature effect on the corrosion inhibition process of CS in 1M HCl containing PPO, the *PDP* experiments were recorded at different temperatures (298-328 K) with and without optimal concentration of our compound (Fig.7 and 8).

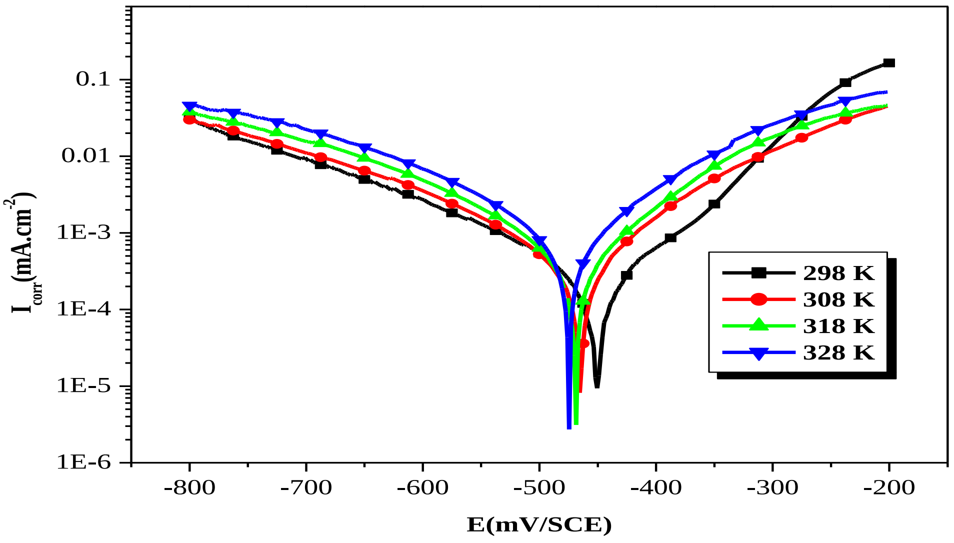

Figure 7. Polarisation plots of CS in 1 M HCl at different temperatures

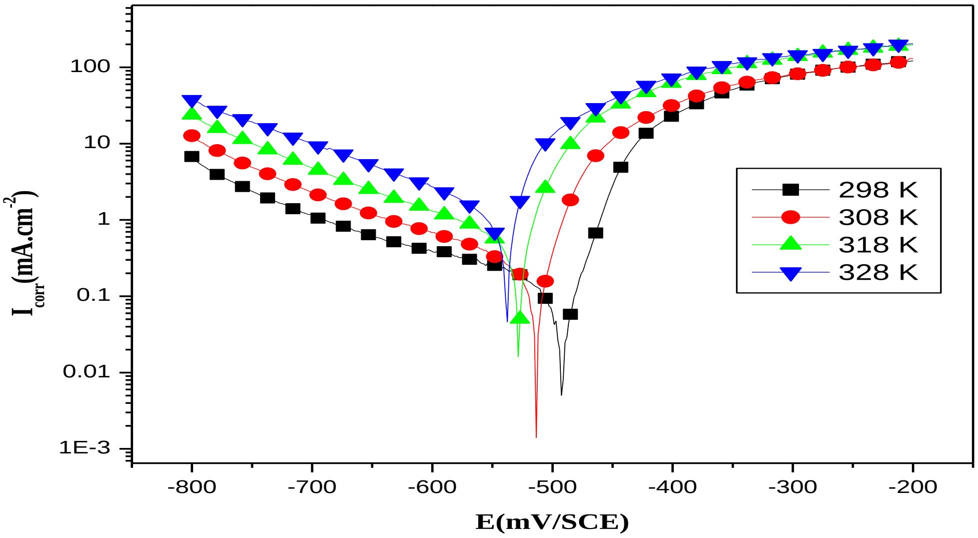

Figure 8. Polarisation plots of CS in 1 M HCl with 10^{-2} M of 6-phenylpyridazin-3(2H)one at different temperatures.

The results are collected and presented in Table 4. In the both environment, *i.e.*, aggressive and protected, the values of I_{corr} are increased with increasing temperature. In uninhibited solutions, more rapidly increase of I_{corr} values is observed. But a nearly constant values of the inhibition efficiency are observed after addition of the **PPO** which indicate the good inhibitive properties of tested compound at higher temperature [33].

Table 4. The results of the temperature effect of steel in acidic medium and with optimum concentration of 6-phenylpyridazin-3(2H)one.

Medium	Temperature (K)	E_{corr} (mV/SCE)	I_{corr} (mA/cm^2)	$-\beta_c$ (mV/dec)	***EI* (%)**
1 M HCl	298	-450	594	186	**----**
	308	-467	896	165	**----**
	318	-470	3428	137	**----**
	328	-477	6720	125	**----**
10^{-2} M of **PPO**	298	-491	107	148	**81.98**
	308	-510	176	147	**80.35**
	318	-528	469	164	**86.31**
	328	-536	1129	171	**83.19**

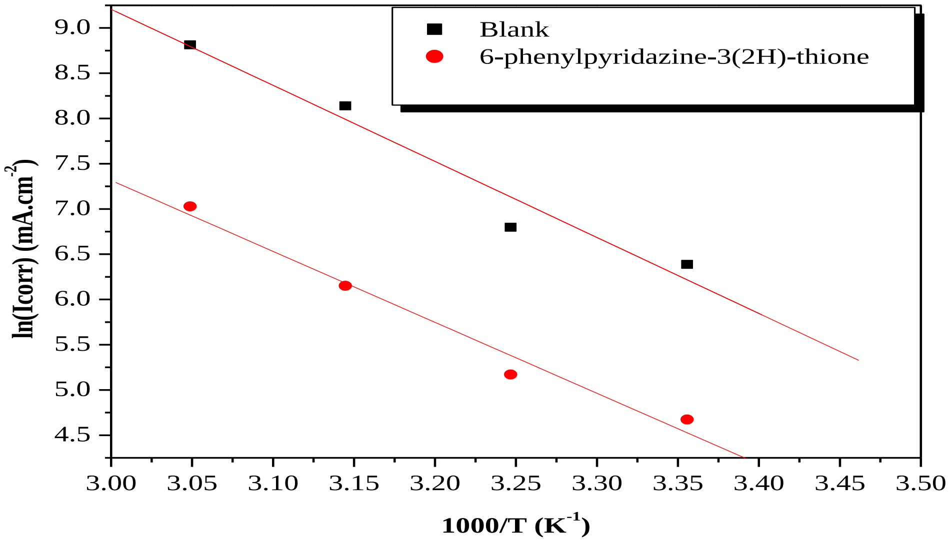

Figure 9. Arrhenius plots of C38 steel in 1 M HCl with and without 10^{-2} M of **PPO**

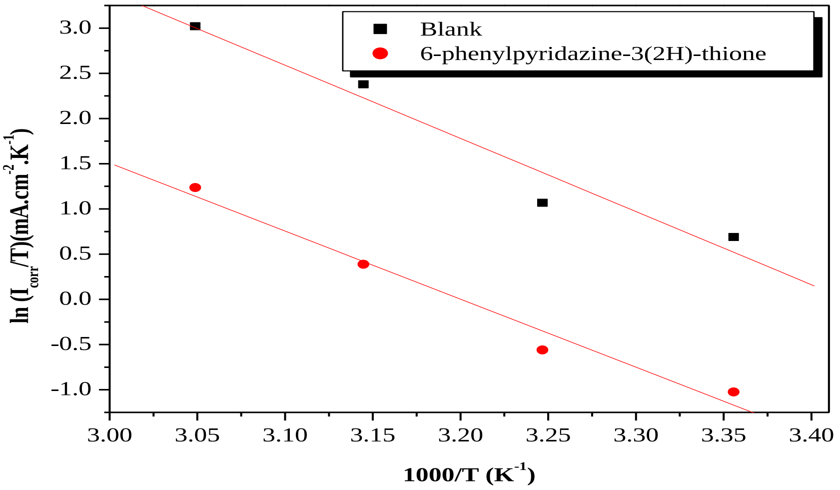

Figure 10. Variation of Ln (I_{corr}/T) versus 10^3/T for blank and 1 M HCl + 10^{-2} M of **PPO**

With the help of the Arrhenius and transition-state plots (Eq. 6-7), the activation parameters for the corrosion process were calculated and presented in Table 5 [33]

$$I_{cor} = \mathrm{A}\exp(-\frac{\mathrm{E_a}}{\mathrm{RT}}) \qquad (7)$$

where I_{corr}, A, E_a and R are the corrosion current density, the Arrhenius constant, the apparent activation energy and the universal gas constant, respectively.

$$I_{cor} = \frac{RT}{Nh}\exp(\frac{\Delta S_a^*}{R})\exp(-\frac{\Delta H_a^*}{RT}) \quad (8)$$

where N, h, ΔS_a^* , and ΔH_a^* are the Avogadro's constant, the Planck's constant, the change of the entropy of activation and the change of enthalpy of activation, respectively.

Table 5. Activation parameters for corrosion process of steel in HCl/**PPO** medium

	E_a (kJ/mol)	ΔH_a (kJ/mol)	ΔS_a (J/mol)	E_a -ΔH_a (KJ/mol)
Blank	69.80	67.22	32.48	2.58
PPO	65.06	62.46	2.70	2.60

The results reveals that the value of E_a in 1 M HCl solution was 69.80 kJ/mol. The addition of 6-phenylpyridazin-3(2H)one to the acid solution decreases the value of E_a which is an indication that the inhibitor is chemically adsorbed on the surface of C38 steel [37-38]. While the positive sign of $\Delta H°_a$ is mainly associated to endothermic nature of the CS corrosion process indicating the slow dissolution of the CS [39] in the presence of 6-phenylpyridazin-3(2H)one. Large value of entropies ($\Delta S°_a$) imply that the activated complex in the rate determining step represents an association rather than a dissociation step [40], meaning that a decrease in disordering takes place on going from reactants to the activated complex [41-42].

4. CONCLUSIONS

It can be concluded as follows:

- The 6-phenylpyridazin-3(2H)one (**PPO**) was found effective inhibition performance against the CS dissolution in acidic solution.
- Polarization studies showed this compound was mixed type inhibitor.
- The presence of 6-phenylpyridazin-3(2H)one increase the inhibition efficiency to reached a maximum value at 10^{-2} M.
- The polarization curves, *EIS* and weight loss measurements were in good agreement.
- Adsorption of inhibitor molecules on the C38 steel surface in inhibited solution followed the Langmuir isotherm.

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